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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

1. **Balances, weights and precise laboratory weighing.** D.S.I.R. (N.P.L. Notes Appl. Sci., 1954, No. 7, 46 pp.).— The design, functioning and adjustment of the various components of the ordinary knife-edge analytical balance are discussed, together with methods for determining the fineness and the sharpness of the knife-edges and carrying out lateral and vertical adjustment of the knives. Brief notes are given on chain, micro, single-pan and other special types of balance, the technique of weighing, and the design, verification and adjustment of weights. Appendices deal with the simple theory of the undamped knife-edge balance, the motion of the damped beam and the effect of air-buoyancy on a single-pan balance. (22 references.) J. M. JACOBS

2. **Effect of the nature of analytically active groups on the sensitivity of analytical reactions.** L. M. Kul'berg and N. I. Davydova (*Dokl. Akad. Nauk. SSSR*, 1954, **95** [3], 551–554).—The sensitivity of a number of benzylidenerhodanine derivatives as reagents for silver or palladium is very considerably increased as the substituent in the *p*-position of the benzylidene group changes from H to $N(CH_3)_2$ and $N(C_2H_5)_2$; this can be explained by the greater polarity (higher dipole moment) of compounds containing the $N(C_2H_5)_2$ group.
R. C. MURRAY

3. **The silver electrode as indicator for hydrogen ions.** H. Khalifa and I. M. Issa (*J. Indian Chem. Soc.*, 1954, **31** [6], 426–430).—To follow up the observation that the potentials set by the silver electrode manifested a tendency to vary with pH of the solution, the silver electrode was applied to the determination of end-points in neutralisation processes. A series of acids, including hydrochloric, nitric, acetic, boric, sulphuric, oxalic, malonic, succinic, tartaric, maleic, citric and phosphoric, were titrated with alkali hydroxides, the titration being followed potentiometrically with the aid of a polished and an aged silver electrode. Curves obtained show a linear potential - pH relationship within the pH range 2 to 9, with a pH gradient amounting to $\simeq$ 21 mV, and indicate that both aged and polished silver electrodes can serve as indicators for hydrogen ions. The end-points obtained agree very closely with those determined with visual indicators. The silver electrode cannot be used to determine pH.
R. J. MAGEE

4. **Anomalies in colorimetric pH measurements.** L. Marcou (*Textilrdsch.*, 1954, **9** [3], 128–141).—Colorimetric pH measurement in solutions is adversely affected by the presence of natural salts, proteins, adsorbents and, particularly, surface-active substances (wetting agents); according to the cationic or anionic nature of the indicator and the impurity present, high or low values are obtained. Surface-active substances cause a change in the state of aggregation and, hence, in the absorption spectrum of the dye molecule. The effect increases in some cases up to a critical concn. of the surface-active agent; above this concn. the molecules of the wetting agent agglomerate, and the indicator molecules become less reactive owing to their peptisation. The use of coloured indicators in presence of surface-active agents should therefore be avoided.
J. TEXTILE INST.

5. **A simple method of measuring rH [Clark].** R. Chini (*Ann. Chim., Roma*, 1954, **44** [3–4], 247–254).—An improvement on Vles and Gex's method (*Compt. Rend.*, 1943, **217**, 601) is described. The potential of the system; glass electrode|solution of known rH|platinum is first measured (E_1), and then that of a similar system, with the standard solution replaced by one whose rH is to be measured, (E_2); the rH of the test solution, rH_x, is calculated from the equation—

$$rH_x = rH_{st} + (2/D)(E_2 - E_1), \text{ where}$$

$2/D = 34{\cdot}4$ at 20° C. The solution of known rH is prepared by adding quinhydrone to a solution having oxidation - reduction properties that would not upset the equilibrium (H_2 + quinone $\rightleftharpoons$ quinhydrone) and of pH less than 8; the rH values of such solutions are independent of pH (below pH 8) and have been tabulated for a series of temperatures (10° to 39° C). Two methods of using an ordinary pH meter for these measurements are described.
R. C. MURRAY

6. **Analysis for industry.** T. S. West (*Ind. Chem.*, 1954, **30**, 231–234).—Methods of determining Ca, Sr, Mg, Zn, Mn, Hg, Th, Cd, Co, Cu, Ni, Al, Tl, Bi, Ag and SO_4'' by titration with ethylenediaminetetra-acetic acid are reviewed with special reference to the indicators used. The applications of indicators for non-aqueous media, and redox and miscellaneous indicators are described. (20 references.)
D. R. PECK

7. **Separation and characterisation of cations precipitated by hydrogen sulphide in an acid medium.** P. Blanc and J. Racine (*Chim. Anal.*, 1954, **36** [10], 272).—The use of $(NH_4)_2S$ in separation of the acid sulphide group metals is replaced by a simpler technique in which stronger HCl (1 + 1) is used for dissolving some of the sulphides. D. R. GLASSON

8. **Photometric titrations.** A. L. Underwood (*J. Chem. Educ.*, 1954, **31** [8], 394–397).—The advantages of photometric titrations are discussed with regard to availability of apparatus, relation of absorbancy to concentration, accuracy and versatility. The applications are discussed, in a general way, in respect of acid - base titrations, oxidation - reduction titrations, turbidimetric titrations and titrations involving complex formation. A wide range of examples is given, particular stress being laid on the value of ethylenediaminetetra-acetic acid as the chelating agent in titrations involving complex formation, *e.g.*, in titration of Cu, Ni, Fe and Bi - Pb or Bi - Cu mixtures.
G. HELMS

9. **High-frequency titrations involving chelation with ethylenediaminetetra-acetic acid. I. Chelation studies.** R. Hara and W. West (*Anal. Chim. Acta*, 1954, **11** [3], 264–274).—The Sargent Model V Oscillometer is applied to the study of the behaviour of ethylenediaminetetra-acetic acid with metallic ions. Titration curves obtained under various conditions indicate the chelate-forming tendencies of the metals and the constitution of the chelates obtained. The results are in accord with those previously obtained by other methods of investigation. W. C. JOHNSON

10. **Near infra-red spectroscopy. I. Spectral identification and analytical applications.** W. Kaye (*Spectrochim. Acta*, 1954, **6** [4], 257–287).—The near infra-red region contains many absorption bands concerned primarily with hydrogenic (⋮CH, :NH, ·OH) stretching vibrations. The more characteristic of these bands are identified and assigned for nearly all the overtone and combination bands, of $CHCl_3$, $CHBr_3$, CH_2Cl_2 and CH_2Br_2 in the region 0·7 to 3·5 μ. The spectra of benzene, methanol and *m*-toluidine are also examined. For the halogens all possible combinations can be accounted for as resolved bands or as background absorption. The selection rules and anharmonicity corrections are readily applicable to the near infra-red region. Vibrations mainly localised in two mol. groups within a given mol. do not couple to produce intense combination bands unless they share a common atom or are connected through multiple bonds. A Colthup-type chart giving position, range, and intensity of characteristic absorption bands is given, and factors perturbing the position and intensity of bands are discussed. A formula is given for the prediction of the lower limit of detectability of a compound as a function of component absorptivities and instrument noise, and the limit of detectability for a number of typical mixtures is given. Precision is 0·004 to 0·5 per cent. when the compound is characterised by ·OH, :NH or ⋮CH groups. (106 references.) A. J. MEE

11. **Emission spectroscopy of organic substances with the aid of electron collision excitation in the glow discharge. II.** S. Schüler and L. Reinebeck (*Spectrochim. Acta*, 1954, **6** [4], 288–301).—The results of emission spectroscopy of organic substances in the low-pressure discharge show that the lines correspond not to transformations between highly excited states of the molecules introduced but to their fragmentation. The main problem in this type of spectroscopy is to identify the fragments. Free radicles may be formed, and these may combine to form new substances with other fragments and with themselves. When relatively high excitation energies are used, unexpected substances may result, *e.g.*, with a series of aromatic compounds diacetylene was produced. At lower excitation energies a number of benzene derivatives give rise to toluene, benzene, etc. Experiments with benzaldehyde showed that the effect of impurities may be amplified. A. J. MEE

12. **Chromatography** (*Brit. Med. Bull*, 1954, **10** [3], 161–250).—This survey of the scope, principles, practice and applications of chromatography from the biochemical and medical aspects includes the following 17 review papers. (1) **Introduction** (A. J. P. Martin, pp. 161–162). (2) **General principles of chromatography** (R. J. P. Williams, pp. 165–169). Early experiments; partition chromatography and elution analysis; frontal and displacement analysis; carrier displacement and gradient elution analysis. 20 refs. (3) **Gas-liquid chromatography** (A. T. James and A. J. P. Martin, pp. 170–176). Apparatus and techniques for the identification and analysis of volatile materials including aliphatic carboxylic acids, aliphatic and aromatic amines, hydrocarbons, alkyl chlorides, alcohols, esters and ketones. 7 refs. (4) **Practical aspects of paper chromatography** (R. Consden, pp. 177–182). Materials and apparatus; methods of preparing paper chromatograms; location of spots and identifications. 22 refs. (5) **Quantitative determination of amino-acids** (A. C. Chibnall, pp. 183–186). One- and two-dimensional paper chromatography; column chromatography on starch, on ion-exchange materials and of dinitrophenyl-amino-acids. 52 refs. (6) **Inorganic chromatography** (F. H. Pollard, pp. 187–191). Ion exchange, alumina, cellulose and paper chromatography; selective reactants; schemes of analysis and quantitative determinations. 11 refs. (7) **Chromatography of organic acids** (J. W. H. Lugg, pp. 192–195). Types of chromatograms; theoretical considerations; applications to fatty and other carboxylic acids. 46 refs. (8) **Chromatography of peptides** (P. N. Campbell and T. S. Work, pp. 196–201). Partition chromatography of peptides and peptide derivatives on paper and starch; adsorption chromatography on silica and charcoal; counter-current methods; ion-exchange methods involving displacement or elution development. 81 refs. (9) **Separation of carbohydrates and phosphoric esters on paper chromatograms** (F. A. Isherwood, pp. 202–210). Mechanism of carbohydrate separation and movement of sugars; purification of filter-paper; solvents; detection and spray reagents; quantitative analysis. 59 refs. (10) **Porphyrins** (J. E. Falk, pp. 211–213). Adsorption and partition column chromatography; paper chromatography. 19 refs. (11) **Chromatography of nucleotides and related substances** (R. Markham, pp. 214–217). General procedure with filter-paper; solvents; detection of the spots. 55 refs. (12) **Thyroid hormones** (J. Gross, pp. 218–223). Advances in thyroid physiology through the application of chromatography; paper and column chromatographic methods. 46 refs. (13) **Antibiotics and vitamins** (T. S. G. Jones, pp. 224–228). Penicillins, streptomycins, polypeptide antibiotics and polymyxins; survey of methods for vitamins; vitamin B_{12}. 57 refs. (14) **Chromatography of steroids and sterols** (I. E. Bush, pp. 229–236). Special aspects and useful techniques; adsorption, partition and miscellaneous methods; detection, characterisation and identification; quantitative methods. 117 refs. (15) **Chromatography of proteins** (R. R. Potter, pp. 237–240). Adsorption, ion-exchange, partition and paper-chromatographic methods. 27 refs. (16) **Separation of amino-acids and lower peptides by displacement chromatography by the use of ion-exchange resins** (S. M. Partridge, pp. 241–246). Displacement development; properties of resins in relation to displacement chromatography; separation of amino-acids from protein hydrolysate and from biological extracts. 26 refs. (17) **Amino-acid metabolism** (C. E. Dent and J. M. Walshe, pp. 247–250). Displacement and elution chromatography on ion-exchange resins; renal and "overflow" amino-acidurias; phenylketonuria. 59 refs. W. H. C. SHAW

13. **Partition chromatography: an isentropic separation process.** A. Klinkersberg (*J. Chem. Educ.*, 1954, **31** [8], 423).—Theoretical treatment of entropy considerations involved in the process of partition chromatography under ideal conditions leads to the conclusion that the separation is not a

reversal of a diffusion process but is an isentropic separation. The reasoning used is analogous to that used in the derivation of the entropy of gaseous mixtures with the aid of semi-permeable membranes.
G. HELMS

14. **Chromatographic analysis with butyryl-, benzoyl- and phthaloyl-cellulose paper[s].** F. Micheel and P. Albers (*Mikrochim. Acta.*, 1954, [5], 489–493).—Chromatographic papers in which the cellulose is partially esterified are prepared from Whatman No. 1 paper. The paper is pre-treated with benzenesulphonic acid in acetic acid and then uniformly and rapidly esterified. The resulting papers contain 16 per cent. of butyryl, 40 per cent. of benzoyl or 14·5 per cent. of phthaloyl groups, and the fibre structure is retained. Compared with acetylcellulose paper, these papers give higher R_F values in the separation of various mixtures. In the phthaloyl paper, only one of the carboxylic groups is esterified and it can be used as an ion-exchanger for the separation of amino-acids and other basic substances; being insoluble in alkalis, it is superior to carboxylcellulose paper.
E. HAYES

15. **Ion-exchange chromatography.** R. P. Shukla and R. P. Bhatnagar (*Agra Univ. J. Res.*, 1954, **3**, 9–12).—The use of sulphonated coal for the effective separation of Ag and Cu in column chromatography is described.
E. G. BRICKELL

16. **"Streaming" in paper electrophoresis.** K. Stolta, S. Brill and A. Ballester (*Hoppe-Seyl. Z.*, 1954, **296** [3–4], 141–147).—Each protein molecule in paper electrophoresis is subject to three actions: (*i*) electro-osmosis, which with Whatman No. 1 paper is so small that it can be neglected, (*ii*) the actual separating effect of the applied current itself, and (*iii*) a "streaming" effect of the buffer, which may increase or decrease the mobility and varies with the current strength. The "streaming" is due to evaporation from the paper; at 6° C it is greater in the first hr. than at 25° C, although the converse is true subsequently. If 0·25 mA per cm of width is used, the degree of "streaming" after 10 hr. is about the same at either temperature. As this period or not much more is required for maximum separation, no advantage is to be gained from conducting the electrophoresis in the cold.
F. POWELL

17. **The staining of paper electrophoretograms with Amido Schwarz.** Z. Pučar (*Hoppe-Seyl. Z.*, 1954, **296** [1–2], 62–66).—The dyeing of proteins on filter-paper and the elution of the dye are discussed with particular reference to the use of Amido Schwarz 10 B. Full details are given for carrying out the electrophoresis as well as the dyeing, washing and elution processes, which are done at 80° C in order to speed them up. A 1 per cent. soln. of the dye in 10 per cent. aq. acetic acid is used, and 5 per cent. aq. acetic acid is used as washing fluid. Protein-bound dye is eluted for quant. measurement with 50 per cent. methanol containing 5 ml of *N* NaOH per litre. At 500 to 600 mμ, extinction values of bound dye are claimed to be proportional to protein concn. but binding power of albumin and globulin are shown to be different. Correction factors are not used in routine analyses, but comparison is made with normals treated similarly to the unknowns.
F. POWELL

18. **Polarographic analysis of pyrrhotite. II. A study of routine analyses.** S. Durovič (*Chem. Listy*, 1953, **47** [7], 999-1002).—With pyrrhotite as an example, the author presents a time study of routine polarographic analyses with respect to their accuracy.
G. GLASER

19. **Polarisation titrations. I. Principle, execution and application.** U. F. Franck (*Z. Elektrochem.*, 1954, **58** [5], 348–354).—The polarisation resistance at a suitable indicating electrode changes sharply at the equivalence point of a titration, if at least one of the ionic species involved determines the rate of the electrode process. This form of titration can be applied to most common titrations, including determination of water with Karl Fischer's reagent. The resistance is measured on low-frequency a.c.; its great concentration sensitivity enables the procedure to be used for very dilute solutions.
R. C. MURRAY

20. **Utilisation of polarisation curves in analytical chemistry.** J. Coursier (*Ann. Chim.*, 1954, **9** [7–8], 353–397).—A general account is given of polarisation curves and a description of methods by which they can be applied to the analysis of various redox systems. The bibliography contains 42 entries.
H. F. W. KIRKPATRICK

21. **Differential thermal analysis technique and its applications.** V. S. Ramachandran and S. K. Bhattacharyya (*J. Sci. Ind. Res., A., India*, 1954, **13** [8], 365–372).—A number of applications of the differential thermal analysis technique in the study of structural changes occurring in solids during thermal treatment are reviewed. Although mainly used in the field of ceramics, the technique has been satisfactorily applied to the study of physical and chemical changes in various other solid substances, including radioactive materials, and in the determination of heats of reaction. (106 references.)
G. C. JONES

22. **Separation and measurement of metals deposited by electrolysis on a mercury cathode.** B. B. Radak (*Bull. Inst. Nuclear Sci., Belgrade*, 1954, **4**, 55–56).—A method proposed for the separation and measurement of metals deposited by electrolysis on a mercury cathode involves washing the Hg with 5 per cent. HNO_3, and polarographic measurement. The method has been compared with the conventional mercury-distillation method by means of Cd, and results of 96 to 97 per cent. and 40 to 89 per cent., respectively, of the quantity electrolysed have been obtained. This disparity is due to distillation of some Cd with the Hg.
L. F. TAYLOR

23. **The physics of particle-size analysis.** (*Brit. J. Appl. Phys.*, 1954, Suppl. No. 3, 218 pp.).—This report of a conference arranged by the Institute of Physics contains 33 papers on different aspects of particle-size analysis. The subjects covered include the relative motion of particles and fluids, the scattering and absorption of light by particles, the significance of shape factors in particle-size analysis, and the visual and automatised counting and sizing of microscopic particles. See also Abstracts 24, 25, 50, 224).
N. E.

24. **Survey of the automatic counting and sizing of particles.** W. H. Walton (*Brit. J. Appl. Phys.*, 1954, Suppl. No. 3, s121–s125).—The theory used in automatic counting and sizing devices is presented, and some of the practical difficulties met with are discussed. Recent developments in the design of spot- and slit-scanners, guard-spot techniques for both counting and sizing, intercept-height and arrested-spot procedures, as well as electronic

memory machines are described briefly. Recommendations are made regarding control of unwanted particles and of optical density in a sample, method of scanning (especially for high-frequency spot-scanners), brightness of specimen, signal-to-noise ratio in the photo-cell circuit, and resolution and depth of focus. An automatic scanner measuring accurately particles as small as 1 μ would involve intermediate photography, in which enlarged photographic images enable the optical factors (resolution, contrast, brightness) to be evaluated separately from the electrical and mechanical factors.

W. J. Baker

25. **A comparison of methods for particle-size analysis.** B. A. Jarrett and H. Heywood (*Brit. J. Appl. Phys.*, 1954, Suppl. No. 3, s21–s28).—Results of comparative tests on apparatus for particle-size analysis over the range of 12 to 53 μ are reported, and recommendations are made for effecting rapid routine analyses of coarse dusts (20 to 76 μ) by sedimentation. Distilled water containing 0·1 per cent. of sodium hexametaphosphate is used to produce, for most samples, a 0·01 suspension of the dust (*i.e.*, $d = 0{\cdot}01$ g per cu. cm higher than that of the pure dispersing liquid). Referred to the specially graded silica test-dust used, the applicable particle-size ranges and probable degrees of error (per cent.) for the various methods examined are: Andreasen pipette, 0 to 45 μ, $\pm$ 0·5; hydrometer, 0 to 25 μ, $\pm$ 1·5; glass-"diver," 0 to 30 μ, $\pm$ 1; roof-"diver," 10 to 53 μ, 0 to $-1{\cdot}5$; closed manometer, 8 to 53 μ, $\pm$ 1·5; photo-extinction, 0 to 25 μ, $\pm$ 3; electrical resistance method, 15 to 40 μ, $\pm$ 3. The decantation and open-manometer methods are unreliable. The newly developed roof-diver method, which is very simple to operate, involves fitting stationary brass divers at the base of a vertical column immersed to a known depth in the suspension; the diver falls from the column when the density of the ambient suspension becomes less than that of the diver. If only a very small sample of dust is available, use of photo-extinction equipment is obligatory, although there is an error of $\pm$ 6 per cent. over the range 25 to 53 μ. Electrical resistance apparatus in its present form is regarded as unsuitable. The roof-diver and closed-manometer methods can be used for sedimentation of $-$ 200 to $+$ 300-mesh fractions, provided the sampling depth is increased to 60 cm.

W. J. Baker

26. **Rapid particle-size distribution analysis.** F. S. Eadie and R. E. Payne (*Iron Age*, 1954, **174**, 99–102).—An instrument known as the Micromerograph for the rapid analysis of particle-size distribution in the range from 1 to 250 μ has been developed by the Sharples Corp., of Bridgeport, Pennsylvania. A sample of the powder is released by nitrogen under known pressure from a special powder feed system incorporating a de-agglomerator. The sample forms a cloud, which settles in a sedimentation column, 7 ft. 4 in. high and 3¾ in. in diam. At the bottom of the column the particles fall on to the pan of a sensitive recording balance. A cumulative weight curve plotted against particle diameter is obtained directly from a recorder chart.

Light Metals Bull.

27. **Radioactive determination of gas flow in large ducts.** E. W. Voice, E. B. Bell and P. K. Gledhill (*J. Iron & Steel Inst.*, 1954, **177** [4], 423–427).—As Pitot-tube surveys are not always convenient for determining the gas flow in large ducts and chambers operating at high temperatures and various pressures, a radioactive tracer method has been devised, in which a known amount of radon is introduced suddenly into the duct and gas samples are taken at regular time intervals at a position downstream from the insertion point. The gas flow is calculated from their activities. The introduction and sampling points must be chosen carefully to ensure intimate mixing of the radon with the gas to be measured. Gas volumes can be determined to an accuracy of $\pm$ 5 per cent.

C. J. Keattch

28. **Dimethylglyoxime.** British Drug Houses, Ltd. (*Lab. Pract.*, 1954, **3** [10], 419–424).—The physical and chemical properties of dimethylglyoxime are reviewed, and the formation and properties of its complexes with Ni, Co, Cu, Pd, Rh and Au and its reactions with several organic compounds are indicated briefly. (130 references.)

S. C. Jolly

29. **Structure and behaviour of organic analytical reagents. VI. Heats and entropies of formation of several divalent metal chelates of 8-hydroxy-2-methyl- and 8-hydroxy-4-methylquinoline[s].** W. D. Johnston and H. Freiser (*Anal. Chim. Acta*, 1954, **11** [3], 201–213).—The Calvin - Bjerrum titration technique (*J. Amer. Chem. Soc.*, 1952, **74**, 1383) for the determination of chelate formation constants is applied to the Cu^{II}, Ni^{II}, Co^{II}, Zn^{II} and Mn^{II} chelates of 8-hydroxy -2-methyl- and -4-methyl-quinolines. Measurements are made at several temperatures in order to evaluate heats and entropies of chelate formation. For all the metals, the heats of formation of the chelates of the 2-methyl compound are more positive than those of the corresponding chelates of the 4-methyl compound. The large difference in the strengths of the metal - chelate bonds is apparently due to the hindrance of the methyl groups in the former reagent. The results are also discussed in terms of the Ni^{II} chelates, which, contrary to the general rule, are less stable than the Co^{II} and Zn^{II} chelates. Some formation constants have also been determined for the chelates of 8-hydroxy-2-phenylquinoline.

W. C. Johnson

30. **Use of thiosemicarbazones in inorganic analysis.** S. S. Guha Sircar and Satyabadi Satpathy (*J. Indian Chem. Soc.*, 1954, **31** [6], 450–454).—Thiosemicarbazones of 8 aldehydes and 5 ketones were tested as analytical reagents for all the common metal ions at various pH values. The solubilities of the precipitates were investigated. In all cases Ag, Hg and Cu formed coloured precipitates and Co a red solution. Quantitative determinations of Hg and Cu can be carried out with the thiosemicarbazones of dihydroxybenzaldehyde (**I**), *p*-dimethylaminobenzaldehyde (**II**) and cinnamaldehyde (**III**). **III** gives the best result, but **II** is highly sensitive for Hg, yielding a yellow ppt. or colour, sensitive at dilutions of 1 in 10^6. The compound of **I** of m.p. 213° C gives a deep red colour with Co in NH_4Cl and aq. NH_3 solution (sensitivity of 1 in 10^8) which can be used for the colorimetric determination of Co.

R. J. Magee

31. **Analytical applications of complexones.** F. Bermejo Martínez (*Inf. Quím. Anal.*, 1954, **8** [4], 119–132).—The theoretical principles underlying the use of complexones in analysis are summarised and analytical applications are reviewed. (37 references.)

L. A. O'Neill

32. **Stability of solutions of sodium tetraphenylboron.** G. H. Gloss and B. Olson (*Chemist Analyst*, 1954, **43** [3], 70–71).—In relation to sodium tetraphenylboron for use in Kohler's method (*Brit. Abstr. C*, 1953, 338), it is suggested that the pH of solutions of the reagent should be adjusted to $\simeq 5$, and that the solution should be stored at or below room temp. and filtered before use. The type of container used is unimportant. H. P. PAGET

33. **Reliability of calculations based upon the law of chemical equilibrium.** D. D. DeFord (*J. Chem. Educ.*, 1954, **31** [9], 460–464).—The law of chemical equilibrium is often used as the basis for the theoretical treatment of an analytical problem. In evaluating the reliability of such calculations the four questions to be answered are: (*i*) does an equilibrium actually exist, (*ii*) are there complicating secondary processes or side reactions causing multiple equilibria, (*iii*) are the known data sufficiently accurate and precise, and (*iv*) are activity corrections necessary or possible? The effects of these factors are discussed, and a number of examples illustrating errors that arise in making calculations based on the equilibrium law are given, *e.g.*, solubility of Ca oxalate in ammonium oxalate, and solubility of AgCl as a function of Cl' and $Ag^{\cdot}$ concentration. G. HELMS

34. **The statistical significance of very low counting rates.** E. A. C. Crouch (A.E.R.E. Rep. C/R 1458, 1954, 7 pp.).—The method of Garwood (*Biometrika*, 1936, **28**, 247) in the application of fiducial limits to events obeying the Poisson distribution is extended to counting problems especially in cases of low total activity, when background count is of same order as activity, *e.g.*, counting of α-particles in the urine of those exposed to radiation hazards. A formula is derived by use of which a series of values of upper and lower limits of mean count at a stated probability level can be given in a table for observed counts between 0 and 150 in number. S.C.I. ABSTR.

See also Abstract 50.

2.—INORGANIC ANALYSIS

35. **Determination of hydrogen by slow combustion over platinum in excess of oxygen.** S. Schuhmann and M. Shepherd (*J. Res. Nat. Bur. Stand.*, 1954, **53** [2], 77–80).—The accuracy and reproducibility of the determination of H by slow combustion with excess of O over a hot platinum wire is investigated, by use of very pure H. The results give the stoicheiometric relationship: $H_2 + 0{\cdot}4996\,O_2 \rightarrow 1{\cdot}4996$ (volume) contraction. When the ideal relationship is corrected for the non-ideality of H and O, it becomes $H_2 + 0{\cdot}4994\,O_2 \rightarrow 1{\cdot}4994$ contraction. The purity of the H calculated from the contraction is 99·98 ± 0·03 per cent., and from the O consumed 99·91 ± 0·13 per cent. When corrected for non-ideality, the values become 100·02 ± 0·03 per cent. and 100·03 ± 0·13 per cent., respectively. J. H. WATON

36. **The determination of deuterium oxide by infra-red spectrometry.** J. Gaunt (*Analyst*, 1954, **79**, 580–585).—A method is described for the analysis of heavy water containing > 99·5 per cent. w/w of D_2O. Heavy water at this concn. is assumed to consist only of D_2O and HOD; the absorption of the HOD is measured at 3 μ. To improve the accuracy of observation, the measurements are made differentially. The optical density of a standard sample is determined and then the optical density of a suitable piece of soda-glass, the ratio of the two observations being recorded. A series of such ratios determined with samples of D_2O of known composition (99·64 to 99·79 per cent.) establishes the calibration graph. The ratio for the unknown sample is then determined in the same way. A similar method is described for determination of low concn. of D_2O (0 to 0·8 per cent.), the other point of fundamental absorption of HOD (3·98 μ) and a cell of distilled water in place of the glass standard then being used. A. O. JONES

37. **Sedimentation method for potassium determination.** D. Kaplan and J. Schnerb (*Bull. Res. Counc. Israel*, 1954, **3** [4], 448–451).—Solutions (4 ml) to be analysed for K are placed in a cylindrical 10-ml centrifuge tube, graduated to 0·2 ml, which has a ground joint to take a funnel-like upper tube. A conc. solution of $Al_2(SO_4)_3$ (sp. gr. 1·345 at 25° C, 15 ml) is added and the mixture is stirred. After 10 min. the clear supernatant liquid is removed with a pipette, and the lower tube is centrifuged for 10 min. at 3000 r.p.m. (not with an angle-head). The volume of pptd. potash alum is proportional to the concn. of K in the original solution over the range 12·5 to 250 g per litre, irrespective of the presence of Na. Ca can first be removed by addition of Na_2SO_4 and filtration. C. E. SEARLE

38. **The photometric determination of copper with ethylenediaminetetra-acetic acid.** W. Nielsch G. Böltz (*Z. anal. Chem.*, 1954, **143** [1], 1–12).—Copper can be estimated both in faintly acid and alkaline solutions with ethylenediaminetetra-acetic acid (**I**). Separate calibration curves are required and the extinction, which is independent of pH between pH 4·75 and 6·50 and almost independent of pH around pH 10, is measured at about 720 mμ, the absorption curve being fairly flat around this wavelength. Neither NO_3', Cl', Br', SO_4'', acetate, borate, tartrate nor $NH_4^{\cdot}$, $Na^{\cdot}$ and $K^{\cdot}$ interfere, and a wide choice of acids for dissolving the sample and buffers is available. By correct choice of conditions, 0·035 to 8·0 mg of Cu per ml can be determined. The method is suitable for the determination of Cu in Sn- and Ni-containing alloys, and it should be free from interference as many metals form water-soluble compounds with **I**.
P. S. STROSS

39. **The colorimetric determination of copper with organic reagents in forensic chemical investigations.** A. L. Shinkarenko, E. A. Gryaznova and L. A. Podkolzina (*Aptechnoe Delo*, 1954, **3** [4], 21–24).—Copper in biological materials is determined by means of diethyldithiocarbamate. The organic matter in a 5 to 25-g sample is first destroyed by the method of Stepanov; Cu (together with Ni and Co) is pptd. from a solution of the residue by means of rubeanic acid; the ppt. is incinerated with H_2SO_4 and the residue is taken up in HCl; the Cu is then determined by forming the diethyldithiocarbamate complex, which is extracted with $CHCl_3$ for colorimetric measurement. Recoveries of 83 to 90 per cent. were attained on samples to which known amounts of Cu had been added. E. HAVES

40. **The heterometric micro-determination of copper with quinaldic acid in the presence of foreign metals.** M. Bobtelsky and B. Graus (*Anal. Chim. Acta*, 1954, **11** [3], 253–260).—The heterometric method (*Anal. Abstr.*, 1954, **1**, 1212) is

applicable to the determination of Cu^{II} with Na quinaldate over a wide range of acidity and in the presence of tartrate and citrate. Titrations of $CuSO_4$ (5 ml of 0·006 *M* soln.) with 0·02 Na quinaldate in the presence of 2 ml of a *M* solution of $Ca(NO_3)_2$, $MgSO_4$, $NiCl_2$, $Co(NO_3)_2$, $AlCl_3$ or $Cr(NO_3)_3$ or 1 ml of *M* $MnSO_4$, show a max. error of − 2 per cent. The permissible concn. of Ba, Zn or Pb is restricted. Interference from $FeCl_3$ (1 ml of *M* soln.) is prevented by the addition of sodium tartrate.

W. C. JOHNSON

41. **The absorptiometric determination of minor amounts of copper in metals.** H. J. Cluley (*Analyst*, 1954, **79**, 561–567).—With ethylenediaminetetraacetic acid (**I**) as masking agent, Cu can be selectively extracted as the complex with diethyldithiocarbamate (**II**). In presence of **I**, only Ag^{+}, Hg^{++}, Pb^{++}, Cd^{++}, Bi^{+++} and Cu^{++} form derivatives with **II**, and of these only the derivatives of Cu and Bi are coloured, so that by solvent extraction with subsequent absorptiometric determination only Bi interferes. In the method described, the sample is dissolved in dil. HNO_3, treated with the reagent **I** (50 g of the disodium salt in 1 litre of water), and excess of aq. NH_3, which is then extracted with a soln. of diethylammonium diethyldithiocarbamate in $CHCl_3$ (10 g per litre). The optical density of the extract is measured in a Spekker absorptiometer with Ilford No. 601 violet filters. The Cu content is ascertained from a previously prepared calibration graph. The absorptiometric measurement includes Bi, if present. After this measurement, the $CHCl_3$ soln. is treated with KCN soln. and the optical density value of the $CHCl_3$ layer is then the result of the Bi only. The method is suitable for the rapid determination of small amounts of Cu in metals. Results are given for Ni - Fe alloys and for five standard samples, *viz.*, 3 steels, an Al alloy and a white metal.

A. O. JONES

42. **Comparison of 8-hydroxyquinoline and 8-hydroxy-2-methylquinoline in the colorimetric determination of copper.** J. Bernal-Nievas and L. Serrano-Berges (*An. Soc. Esp. Fis. Quim., B*, 1954, **50** [5], 459–464).—In chloroform solution, the Cu derivative of 8-hydroxy-2-methylquinoline shows better agreement with Beer's Law than that of the 8-hydroxyquinoline one; the light absorption and the variation of transmission with Cu concentration are greater also. With 8-hydroxy-2-methylquinoline, Al and Mg do not interfere, but with 8-hydroxyquinoline Al interferes; a sequestering agent (Na tartrate) must be used and calibration curves must be constructed according to the amount of agent added.

L. A. O'NEILL

43. **The spectrographic analysis of brass and other materials by the porous-cup method.** L. G. Young, J. M. Berriman and B. E. J. Spreadborough (*Analyst*, 1954, **79**, 551–561).—A method of spectrographic analysis involving the graphite-cup - spark technique of Feldman (*Brit. Abstr. C*, 1950, 108 and 124) has been applied to determination of the major constituents of alloys. The apparatus used is described and illustrated, and details are given of its application to determinations of Zn in brass. Indications are given also of its application to steels and to determining Mg in Fe, Al in Cu, and Al in Mg alloys. The use of added gold as an internal standard in the quant. analysis of soluble residues, corrosion products and ppt. is described.

A. O. JONES

44. **Polarographic and polarometric study of some noble metals. VI. Selective polarographic determination of gold.** J. Čihalík, J. Doležal, V. Simon and J. Zýka (*Chem. Listy*, 1954, **48** [1], 28–31).—A selective polarographic determination of Au is possible by using a 0·5 to 1 *M* soln. of ethylenediamine tartrate and a 0·1 *M* soln. of $Na_4P_2O_7$ as electrolyte. The reduction of Au^{+++} in this medium on the dropping-mercury electrode (*vs.* S.C.E.) is characterised by a diffusion-controlled wave ($E_{\frac{1}{2}} = -0{\cdot}02$ V), its height being a linear function of gold concn. Au can be selectively determined in the presence of Cu^{++}, Bi^{+++}, Fe^{+++}, Pb^{++}, UO_2^{++}, Pd^{++}, Ce^{++++}, Sb^{+++}, Cd^{++}, Ti^{++++}, Mo^{++++++}, Ni^{++}, Te^{++++}, Se^{++++}, W^{++++++} and Zn^{++}. The simultaneous determination of some of these metals is possible if their concentrations are approx. equal. A graph of polarographic spectra is given.

G. GLASER

45. **A rapid spectrographic method for the determination of beryllium in air dust.** F. P. Landis and M. C. Coons (*Applied Spectroscopy*, 1954, **8** [2], 71–75).—The dust in 0·1 cu. metre of air is collected on a small filter-paper of 0·84 in. diam. The rolled filter-paper is inserted in a small cavity drilled in the graphite spectrographic electrode and carbonised *in situ* by blowpipe heating. Five mg of $BaCl_2$ containing 0·1 per cent. of Tl_2O_3 are then inserted into the cavity and mixed with the sample. $BaCl_2$ acts as carrier and Tl is present as internal standard if required. Conventional d.c. arc excitation is used. The sensitivity of the method is 0·002 μg of Be per sample.

B. S. COOPER

46. **The spectrochemical analysis of beryllium in ore, air and solutions.** P. W. Kehres and W. J. Poehlman (*Applied Spectroscopy*, 1954, **8** [1], 36, 38, 40 and 42).—By spark excitation of a briquetted sample, Be may be determined in beryl ore containing 6 to 12 per cent. of BeO. Precision is ± 3 per cent. of content. Briquettes contained 0·375 g of beryl ore (100 mesh), 0·25 g of Fe_2O_3, 0·5 g of Li_2CO_3 and 1·25 g of pure graphite. Fe acts as internal standard and Li as buffer. A suitable line pair is Be 3321·3 A, Fe 3021·1 A. For low concentrations of Be in air or in solution, the method of Cholak and Hubbard (*Brit. Abstr. C*, 1948, 81) has been modified; this has enabled the sensitivity to be extended to 0·00025 μg of Be per ml of solution.

B. S. COOPER

47. **A phosphate titrimetric procedure for the estimation of magnesium, zinc, manganese and cadmium.** P. R. Subbaraman (*J. Sci. Ind. Res., B, India*, 1954, **13** [8], 553–556).—The method of titrating phosphates with bismuthyl perchlorate, by using *s*-di(allylthiocarbamyl)hydrazine in chloroform as indicator, has been extended to the estimation of Mg, Zn, Mn and Cd. These metals are precipitated as their double ammonium phosphates and titrated with bismuthyl perchlorate after solution in dilute perchloric acid. From the phosphate contents of the precipitates, the stoicheiometric equivalents of the metals are computed. Besides being equally reliable, the titrimetric method is simpler than its gravimetric counterpart.

G. C. JONES

48. **The rapid determination of lime and magnesia in soda-lime glasses.** H. J. Cluley (*Analyst*, 1954, **79**, 567–573).—A rapid method for determination of Ca and Mg in soda-lime glasses is described. The sample (0·1 g), ground to pass a 120-mesh sieve, is decomposed with HF and $HClO_4$ and then

ignited at 500° C to remove the last traces of F. The residue is dissolved in HCl under specified conditions, and Fe and Al are extracted with a soln. of 8-hydroxyquinoline in $CHCl_3$. The aq. layer is made alkaline with NaOH, murexide indicator is added and the Ca is titrated with standard disodium ethylenediaminetetra-acetate soln. The corresponding aq. layer from another sample portion is treated with an NH_4Cl - aq.NH_3 buffer soln. and KCN and, after addition of Eriochrome black T indicator, the Ca and Mg are titrated together. Both titrations are corrected for blank results, and the Mg content is calculated from the difference. The method can be applied in the presence of Zn, but not in the presence of Pb or Ba. A. O. JONES

49. **The titration of calcium with ethylenediaminetetra-acetate in the presence of magnesium.** R. D. Bond and B. M. Tucker (*Chem. & Ind.*, 1954, [40], 1236–1237).—Three methods are given for the determination of Ca by titration with disodium ethylenediaminetetra-acetate (**I**). (*a*) To a suitable aliquot soln. add 1 ml of 20 per cent. sucrose soln., 2 ml of 0·2 *M* Na_2CO_3 soln., and one drop of 0·1 per cent. Nile blue soln.; to this mixture add 10 per cent. NaOH soln. dropwise until a pink colour appears, then add 1·0 to 1·5 ml of NaOH in excess followed by 40 mg of unscreened murexide indicator and titrate the mixture with a standard soln. of **I**. Excess of Mg obscures the end-point. (*b*) Add 1 ml of 20 per cent. sucrose soln. and 2 ml of 0·2 *M* Na_2CO_3 soln. to an aliquot portion. and make the soln. up to 80 ml, add 3 to 4 ml of 10 per cent. NaOH in excess of that required to give a pH of 7·0, then 40 mg of screened murexide indicator and titrate with **I**. (*c*) This method is applicable to NH_4Cl soil extracts. Add 2 ml of 0·2 *M* Na_2CO_3 soln. to a neutral aliquot, dilute it to 50 to 100 ml and then add 10 per cent. NaOH according to the amount of NH_4Cl present (5, 10 or 20 mg-equiv. of NH_4^+ require 3, 5 or 10 ml of NaOH soln., respectively). Finally titrate with **I**, using the screened indicator. In all methods NaOH should be added immediately before the murexide indicator and titration completed without delay. The methods are suitable for titrating 0·02 ± 0·001 mg-equiv. of Ca in the presence of up to 2 mg-equiv. of Mg. G. R. WHALLEY

50. **The application of methods of particle-size and surface-area measurement to whiting.** G. E. Bessey and D. C. Soul (*Brit. J. Appl. Phys.*, 1954, Suppl. No. 3, s181–s189).—Particle-size distributions and surface areas of samples of whiting (size-range 0·1 to 200 μ, but chiefly 1 to 10 μ) were determined by standard methods, and the results were compared in respect of a possible evaluation of the practical uses of the powder. Although all methods yield results of the same order, agreement is poor and correlation only moderate. Coarse particles (> 45 μ) can be determined accurately by wet sieving, finer particles (2 to 25 μ) by the Andreasen pipette method and particles from 0·5 to 2 μ by either the Andreasen or centrifugal sedimentation methods, a suspension in water being used in all instances. The lengthy nitrogen-adsorption method gives absolute values of specific surface, *S*, which, however, are not necessarily of practical significance. The simple and rapid air-permeability method measures the effective external surface, the values being generally higher than those calculated from particle-size distribution data, which ignore internal surface, irregularity of surface and shape. When values of *S* are required only for comparing mean particle-size or as a simple index of fineness, the air-permeability method (with Rose's correction) is quite adequate. The true (N-adsorption) values of *S* are approx. equiv. to air-permeability values × 1·6. Values of *S* determined with the Spekker absorptiometer are unreliable. Procedures, methods of calculation and presentation of size-distribution data are described. Log-normal size-distribution in whitings is fairly frequent, geometric mean diameters varying from 2·2 to 6·8 μ with values of *S* (apparent) from 7000 to 14,000 sq. cm per g. W. J. BAKER

51. **Determination of rare metals in blende by spectroscopic and polarographic methods.** G. Canneri and D. Cozzi (*Chim. e Ind.*, 1954, **36**, 354–357).—On a sample of blende (74·4 per cent. ZnS) obtained by flotation, good agreement was obtained between spectrographic and polarographic determinations of Ge (45, 43 μg per g) and Tl (95, 92 μg per g). The contents of Ga (5 μg per g) and In (0·03 μg per g) were much lower and could be determined only spectrographically. L. A. O'NEILL

52. **A micro-analytical method for the determination of boron in coal.** F. H. Kunstmann and J. F. Harris (*J. Chem. Metall. Min. Soc. S. Afr.*, 1954, **55** [1], 12–15).—A volumetric method for the determination of B in coal is described in which the B is separated as methyl borate by a distillation process. Quantities of the order of 10 to 100 μg can be determined, with a variation (between analysts) of not more than 3 μg. G. C. JONES

53. **Determination of boron in titanium alloys by means of ion exchange.** G. Norwitz and M. Codell (*Anal. Chim. Acta*, 1954, **11** [3], 233–238).—A method is presented for the determination of 0·025 to 1 per cent. of B in Ti alloys. The greater part of the Ti is removed from the sample soln. by means of a cation-exchange resin and the remainder by boiling with $CaCO_3$. *Procedure*—Dissolve 2 g of a Ti alloy containing 0·025 to 0·5 per cent. of B, or 1 g of a sample containing 0·5 to 1 per cent. of B, in 20 ml of dil. (1 + 1) HCl by heating under a reflux condenser. To the hot soln., add dil. (1 + 1) HNO_3 until the blue colour disappears, add 1 to 2 drops of dil. HNO_3 in excess and dilute to 150 ml with water. Charge an ion-exchange column of 3-cm diam. with 80 g of Dowex 50 cation-exchange resin free from "fines" and wash the column with dil. (1 + 2) HCl and then with water. Pour the sample soln. through the column and wash through with dil. (1 + 50) HCl. Neutralise the eluate with $CaCO_3$, add several grams in excess, boil the soln. for 30 min. under a reflux condenser, filter and wash the filter with boiling water. Cool the filtrate to 10° to 15°C, add 2 g of mannitol and titrate the boric acid with standard NaOH soln, using phenolphthalein as indicator. Add a further 2 g of mannitol and re-adjust to the end-point if necessary. Correct for the blank value obtained, using a boron-free sample of Ti. The standard NaOH soln. is $\simeq 0{\cdot}05$ *N* and is standardised on B_2O_3 that has been added to pure Ti and submitted to the procedure. The necessity of using boron-free glassware is emphasised. None of the elements found in commercial Ti alloys interferes. W. C. JOHNSON

54. **Use of morin in chemical analysis. II. Volumetric determination of gallium and indium with complexone III.** V. Patrovský (*Chem. Listy*, 1953, **47** [9], 1338–1341).—Gallium and indium are determined by titration with complexone III in the presence of morin; the end-point is indicated by the disappearance of the characteristic fluorescence of the complex of morin with Ga and In in acetic acid

soln. Interference by Al is suppressed by the addition of F' or BF_4', which extinguish the fluorescence of the Al - morin complex. Ga in bauxite and silicates is determined as follows. Fuse the finely-powdered sample (5 to 10 g) with 5 times its weight of Na_2CO_3, extract the melt with hot water, remove any SiO_2, acidify the extract with HCl and evaporate until crystallisation of NaCl begins. Cool, add an equal vol. of conc. HCl, filter off NaCl and $AlCl_3$ crystals. To the filtrate, add a little $Na_2S_2O_3$ to reduce Fe^{III}, and extract with 2 portions of ether. Dry the extract containing $GaCl_3$ over anhyd. Na_2SO_4, evaporate to dryness, mix the residue with Na acetate (2 g), NaCl (2 g) and glacial acetic acid (1 ml), dil. to 50 ml, add 0·1 per cent. ethanolic soln. of morin (0·5 ml) and 2 ml of a soln. prepared by dissolving borax (1·8 g) and NaF (3·2 g) in water, acidifying with acetic acid and diluting to 100 ml. Titrate with 0·01 *M* complexone III in u.v. light. Indium is determined similarly. A detailed method for the determination of indium in sphalerite is given. G. GLASER

55. **Polarography of niobium. I. Reduction in mineral acid media.** S. K. Dhar (*Anal. Chim. Acta*, 1954, **11** [3], 289–296).—Niobium gives a polarographic wave with the characteristics of a catalytic reduction wave. The reaction is favoured by the presence of HNO_3 or KNO_3, but is depressed by Cl', ClO_4' or SO_4''; it is assumed that the wave is due to the reduction of a nitroniobate complex, $[NbO(NO_3)_5]''$. W. C. JOHNSON

56. **The X-ray spectroscopic determination of niobium and tantalum in rare-earth ores.** D. M. Mortimore, P. A. Romans and J. L. Tews (*Applied Spectroscopy*, 1954, **8** [1], 24–28).—By use of the technique of X-ray fluorescence spectroscopy a method has been developed for determining Nb and Ta in ores associated with monazite. To minimise the effects of large amounts of impurity elements, an internal-standard procedure was adopted; Zr and Hf were found to be most satisfactory for this purpose. The lower concentration limit is 0·05 per cent. for Nb and 0·2 per cent. for Ta; the standard deviation is approximately 5 per cent. at concentrations of 27 per cent. of either element.
B. S. COOPER

57. **Spectrographic determination of yttrium, lanthanum and cerium.** D. S. Pešić (*Bull. Inst. Nuclear Sci., Belgrade*, 1954, **4**, 49–53).—The copper - spark method of Nachtrieb *et al.* (*cf. Brit. Abstr. C*, 1948, 28), slightly modified, has been used for the determination of Ce (10^{-5} to 10^{-6} g), Y (2×10^{-7} to 2×10^{-6} g) and La (2×10^{-7} to 2×10^{-6} g). The Ce line of 4151·97 A is not recommended for the quant. determination of Ce, as it coincides with the line La II 4195·65 A.
L. F. TAYLOR

58. **Polarographic half-wave potentials. I. Method of measurement; half-wave potentials of thallium.** A. A. Vlček (*Chem. Listy*, 1954, **48** [2], 189–193).—The three-electrode system (S.C.E., normal calomel electrode, dropping-mercury electrode) with a compensating circuit was used to measure the $E_{\frac{1}{2}}$ values of $Tl^{\cdot}$ in 4 media at 25° C with an accuracy of $\pm$ 1 mV: in 0·1 *M* KCl, − 494·0 mV; in 0·1 *M* $NaNO_3$, − 493·0 mV; in 0·1 *M* Na_2SO_4, − 403·5 mV; in 0·1 *M* NH_4Cl - aq.NH_3, − 501·0 mV. [Abstractor's note: The $E_{\frac{1}{2}}$ value in Na_2SO_4 quoted is given in the body of the text; the author's summary gives it as − 503·5 mV.]
G. GLASER

59. **Polarographic determination of titanium in soap.** J. Hejna (*J. Amer. Oil. Chem. Soc.*, 1954, **31** [8], 350–352).—A polarographic method for the determination of titanium in soap is described. The soap is ashed, fused with $KHSO_4$ and dissolved in *N* H_2SO_4 soln. saturated with sodium oxalate. The cathodic wave of the diffusion current is well defined and is in direct proportion to the concn. of the titanic ions. Comparison of polarographic and colorimetric methods shows excellent agreement for determination of titanium. D. BAILEY

60. **Method for distinguishing rutile and anatase titanium dioxide.** R. S. Dantuma (*Verfkroniek*, 1954, **27**, 64–67, 126).—A stiff paste of the pigment with water is coated on to a glass slide in a layer $\simeq$ 1 mm thick. Light from a u.v. analytical lamp is projected at 45° on to the uncoated side of the slide, and the amount of light reflected at 90° to the surface is estimated from the density of the image produced on a photographic film. The test sample should be exposed between control samples of anatase and rutile TiO_2. Under the conditions of test the amount of reflected light from anatase will be considerably greater than that from rutile, the percentage of light transmitted through the negative after development being 40 to 50 per cent. for anatase and 75 to 85 per cent. for rutile. Comparative percentage transmission for other white pigments under the test are: white lead 13·5, Sb_2O_3 14·5, lithopone 16, $BaSO_4$ 17·5, $CaSO_4$ 20, $CaCO_3$ 28, ZnO 79·5 and lead titanate 86. Significance of the results on the TiO_2 pigments in respect of their chalking tendencies is discussed. The test does not distinguish between grades of rutile TiO_2 with different resistance to chalking. L. A. O'NEILL

61. **2-Amino*iso*phenoxaz-3-one and 2-acetamido-*iso*phenoxaz-3-one as reagents for stannous ions.** E. Ružička (*Chem. Listy*, 1954, **48** [1], 45–48).—A method has been developed for the identification of Sn^{II} that depends on the characteristic colour change produced when a soln. containing Sn^{II} (in a test tube or on filter-paper) is treated with a 0·1 per cent. ethanolic soln. of 2-amino*iso*phenoxaz-3-one [Ring Index] (**I**) or of 2-acetamido*iso*phenoxaz-3-one [R. I.] (**II**) in the presence of strong mineral acid. With **I**, the colour changes from brown-red to pale yellow-green and with **II** from orange-red to pale blue. Ti^{III} produces similar colour changes and, if present, must be converted to Ti^{IV} by air oxidation of the strongly basified soln. Cu^{II} interferes only if present in amounts greater than one-half of that of the Sn^{II} present. G. GLASER

62. **The microchemical electrolytic analysis of alloys in hydrochloric acid solutions. II. White metals and solders.** A. J. Lindsey and E. A. Tucker (*Anal. Chim. Acta*, 1954, **11** [3], 260–263).—The methods of electro-deposition with controlled potential, previously described for brasses and bronzes (*Anal. Abstr.*, 1954, **1**, 2688), are adapted to the analysis of white metals and solders. White metals are treated as follows. Dissolve 5 to 20 mg in aq. HCl with the addition of $KClO_3$ and evaporate to small bulk. Dissolve the residue in 1·5 ml of aq. HCl and 4 drops of 50 per cent. aq. hydrazine hydrate, heat the soln. to 80° C, stir with H or N and electrolyse at 0·75 V. When the current falls to about one-tenth of its original value, increase the voltage by $\simeq$ 0·05 V and continue until the current falls again. Wash, dry and weigh the cathode, as previously described, to determine the wt. of Cu

plus Sb. Replace the cathode in the apparatus and cover it with water containing 7 drops of conc. HNO_3 and 2 drops of 40 per cent. aq. HF. Heat the liquid and add 10 per cent. aq. $K_2Cr_2O_7$ until the yellow colour persists and the deposit is dissolved. Discharge the colour with 50 per cent. aq. hydrazine hydrate and add 3 drops in excess. Electrolyse at 0·7 V for 10 min. to deposit the Cu, and calculate the Sb by difference. Determine the Sn and Zn as in bronzes. Pb is weighed with the Sn. Solders consisting almost entirely of Pb and Sn are analysed for Pb plus Sn as previously described and the Pb is determined as PbO_2. Alternatively, the following procedure is used. Dissolve the alloy (5 to 20 mg) in 3 ml of conc. HNO_3 and 7 drops of 40 per cent. aq. HF. Electrolyse at the b.p. with the inner electrode as anode for 10 min. at 1·0 V. The Pb is deposited as PbO_2. Immerse the electrode in water containing 2 ml of HNO_3 and 5 drops of 40 per cent. HF, boil the soln. and reverse the current to dissolve the deposit. Cool the soln., reverse the current again and deposit the PbO_2 on the inner electrode. Wash, dry and weigh the electrode, and calculate Sn by difference. W. C. JOHNSON

63. **Complexometric titrations (chelatometry). VI. Catechol violet, a new specific indicator. VII. Determination of thorium.** V. Suk, M. Malát and O. Ryba (*Coll. Czech. Chem. Comm.*, 1954, **19** [4], 679–683).—Direct complexometric determination of Th in acid media ($\simeq$ pH 3) with catechol violet (catecholsulphonephthalein) as indicator is described. The selectivity of the determination is good; Th can be determined in the presence of U and a large number of other cations, *e.g.*, Pb, Ni, Nd, Pr, Ce and La. [This is a translation into German of a paper that appeared in *Chem. Listy*, 1954, **48**, 533.] D. R. GLASSON

64. **The determination of phosphate in the presence of soluble silicates. Application to the analysis of basic slag and fertilisers.** H. N. Wilson (*Analyst*, 1954, **79**, 535–546).—The extent of the interference of SiO_2 in the pptn. of P_2O_5 as quinolinium molybdophosphate is investigated. This interference can be overcome by addition of citric acid to form a complex with MoO_3 that reacts with P_2O_5 but not with SiO_2. Details of the application of this principle to the analysis of basic slag are given, the P_2O_5 being finally pptd. as quinolinium molybdophosphate, which is collected and decomposed with a small excess of 0·5 *N* NaOH soln., the excess being back-titrated to thymol blue - phenolphthalein mixed indicator with 0·5 *N* HCl. For basic slag it is claimed that this method is superior to the statutory method. With some slight modifications in detail the method is applicable to the accurate determination of P_2O_5 in fertilisers. A. O. JONES

65. **Combination of poly- and metaphosphates with organic basic compounds.** J.-P. Ebel and J. Colas (*Compt. Rend.*, 1954, **239** [2], 173–175).—The insoluble complexes formed by combination of crystalline $Na_4P_2O_7$ (**I**), $Na_5P_3O_{10}$ (**II**), $Na_3P_3O_9$ (**III**), $Na_4P_4O_{12}$ (**IV**), or Graham's salt with certain organic basic compounds of low mol. wt. are reported. In acid soln. certain aromatic mono- and diamines and hydrazines form such complexes with Graham's salt; at higher pH, 2-naphthylamine, diaminobenzidine, and *o*-tolidine combine with **I**, **II**, and **IV**; benzidine combines with Na_3PO_4 as well. Yields of the insoluble complex are 70 to 100 per cent., although the reaction is quant. with benzidine in presence of ethanol and acetic acid. At pH 2 to 4, Graham's salt forms insoluble complexes with arginine, histidine, lysine, ornithine, hystidylhistidine, histamine, and some deriv. of guanidine; none of these biologically important compounds forms precipitates with **I**, **II**, **III** and **IV**. Preferential pptn. of a specific complex can be effected by close control of pH with HCl or acetic acid; yields are 50 to 80 per cent. in 1 per cent. solutions of the organic base, but may be increased to 80 to 95 per cent. by increasing the concn. of dibasic amino-acid and pptg. in presence of ethanol. The complexes dissociate easily, especially in presence of excess of HCl. The quant. or semi-quant. separation of certain biological basic compounds by the above method is suggested. W. J. BAKER

66. **Analysis for industry. [Complex phosphates.]** D. Haywood (*Ind. Chem.*, 1954, **30**, 447–449).—Methods for analysing complex phosphates are reviewed. Hexametaphosphate, trimetaphosphate, orthophosphate, pyrophosphate and polyphosphate are determined in the presence of each other by selective pptn. of 4 separate aliquots and conversion of each ppt. to orthophosphate for evaluation as ammonium molybdophosphate. Pyrophosphate may be pptd. in the presence of polyphosphate by manganous salts. The existence of tetraphosphate is demonstrated by chromatography, this technique being useful for phosphate separation. Separation by pptn. with $BaCl_2$ is discussed, and a method for determining triphosphate in the presence of polyphosphate with $ZnSO_4$ is proposed. A. M. SPRATT

67. **The photometric determination of bismuth with thiourea. II. In hydrochloric acid solution.** W. Nielsch and G. Böltz (*Z. anal. Chem.*, 1954, **143** [1], 13–17).—The determination of Bi in acid solution, described previously for dil. HNO_3 (*Anal. Abstr.*, 1954, **1**, 2661), can be carried out in dil. HCl. The extinction is independent of the concn. of thiourea and HCl if their concn. are $>$ 8 per cent. and between 2·8 and 7·5 per cent., respectively; it should be measured in the region of 440 mμ, 10 min. after addition of the reagents. The temperature should be maintained constant. P. S. STROSS

68. **Complexometric titrations (chelatometry). IV. Catechol violet as a new specific indicator: determination of bismuth.** M. Malát, V. Suk and O. Ryba (*Chem. Listy*, 1954, **48** [2], 203–206).—A simple highly selective method for the complexometric determination of Bi is based on the use of catechol violet (catecholsulphonephthalein) (**I**) as indicator in media of pH 2 to 4. To the soln. containing Bi, acidified with HNO_3, add a few drops of a 0·1 per cent. aq. soln. of **I**; a clear blue colour should develop. A violet coloration is a sign of too great acidity; the colour should be adjusted to the required blue shade by adding aq. NH_3. Titrate with a standard soln. of complexone III of suitable concn. (*e.g.*, 0·002 *M*); at the end-point the colour changes to yellow after a transient violet. Pb, Cd, Cu, Ag, Al, Zn, Co, Ni, Mn, Ca and Mg may be present without interfering. G. GLASER

69. **Colorimetric determination of molybdenum and uranium with organic reagents.** F. Will, III. (*Dissert. Abstr.*, 1954, **14** [5], 761–762).—Organic reagents for the colorimetric determination of Mo and U are discussed. A new procedure for determining Mo with disodium 1:2-dihydroxybenzene-3:5-disulphonate (Tiron) is applicable to Mo solutions and standard samples of steel and other

materials. Another procedure for Mo determination involves thioglycollic acid and is applicable to determination of Mo in presence or absence of Fe and of Mo and Fe in the same aliquot. A new procedure also is described for U^{VI} determination with dibenzoylmethane. L. F. TAYLOR

70. **Use of complexones in chemical analysis. XLII. Separation of molybdenum and vanadium by means of 8-hydroxyquinoline.** M. Malínek (*Chem. Listy*, 1954, **48** [1], 38–40).—The previously described gravimetric determination of Mo with 8-hydroxyquinoline (**I**) (Přibil and Malát, *Chem. Listy*, 1950, **44**, 97) has been adapted to the determination of Mo in the presence of V. The method makes use of the property of complexone II to form, at a low pH, a stable complex with $VO_2^{\cdot\cdot}$ that is resistant to **I**. The reduction of $VO_3^{\prime}$ to $VO_2^{\cdot\cdot}$ is best and quantitatively effected by boiling with the complexone itself. *Procedure*—Acidify the soln. containing Mo, V and other cations to pH 1 to 2, add solid complexone II and boil for 10 min. If considerable amounts of V are present, carry out the reduction with a few crystals of Na_2SO_3 at pH < 1. After diluting to 100 ml, neutralise the soln. with aq. NH_3 to pH 3 to 5, add buffer (5 ml of a soln. of 50 g of ammonium acetate in 60 ml of glacial acetic acid and 100 ml of water) and precipitate the hot soln. with a 3 per cent. soln. of **I** in dil. acetic acid partly neutralised with aq. NH_3. Filter hot, wash the ppt. with hot water and dry at 150° C. G. GLASER

71. **Use of complexones in chemical analysis. XLI. Colorimetric determination of uranium with dibenzoylmethane.** R. Přibil and M. Jelínek (*Chem. Listy*, 1953, **47** [9], 1326–1332).—A highly selective method of determining U, based on the colorimetric estimation of the yellow uranyl - dibenzoylmethane (**I**) complex, is described. Extract a 20-ml sample containing 0·05 to 0·5 mg of U with a 0·5 per cent. soln. of **I** in ethyl acetate (10 ml) by shaking for 5 min., re-extract the aq. layer with the reagent (15 ml) for a further 10 min., treat the combined extracts with a few drops of ethanol, make up to 25 ml with ethyl acetate and measure the extinction at 410 mμ. If other cations are present, treat the slightly acid soln. of the sample containing < 0·5 mg of U with 5 per cent. soln. of complexone III, bind the excess of complexone by adding 1 per cent. aq. $Ca(NO_3)_2$, adjust to pH 7 with NH_3 and extract with 3 portions (5, 10 and 10 ml) (each for 10 min.) of **I** solution. Anions that form complexes or insol. ppt. with U, *e.g.*, oxalates, tartrates, citrates, $CO_3^{\cdot\cdot}$ and $PO_4^{\prime\prime\prime}$, must be absent. G. GLASER

72. **Colorimetric determination of uranium with ammonium thiocyanate.** V. S. Jovanović and E. F. Zucker (*Bull. Inst. Nuclear Sci., Belgrade*, 1954, **4**, 111–118).—The suitability of the NH_4CNS method, for the colorimetric determination of U in presence of Fe without pH control, has been investigated by examining the influence of H_2SO_4, HNO_3 and HCl on colour intensity and the stability of the uranyl thiocyanate complex. The colour intensity of the uranyl thiocyanate complex depends on the kind and amount of acid present; conc. acids and 4 *N* HNO_3 cannot be used. HCl and HNO_3 react with the thiocyanate ions and increase the absorption of light in the solution, whilst H_2SO_4 gives a less intense colour, but more reproducible results. The thiocyanate method is applicable to U determination without pH control in an H_2SO_4 or HCl solution with controlled amounts of acid, $SnCl_2$ and NH_4CNS. L. F. TAYLOR

73. **Polarographic determination of uranium.** J. Doležal and J. Adam (*Chem. Listy*, 1954, **48** [1], 32–37).—The polarographic behaviour of U in NaOH and $(NH_4)_2CO_3$ in the presence of 1:2-dihydroxybenzene-3:5-disulphonic acid (Tiron) was studied. In the solutions of Tiron in NaOH, the reduction of U^{VI} occurs at − 1·1 V, corresponding to a one-electron change U^{VI} to U^{V}. In $(NH_4)_2CO_3$ solutions containing Tiron, U forms a complex reduced at − 0·6 V (a two-electron change U^{VI} to U^{IV}). The $(NH_4)_2CO_3$ - Tiron electrolyte is suitable for the determination of U in the presence of Fe. Interference by Pb and much Cu is masked by the addition of complexone III and KCN; in this medium, U is reduced in 2 waves with $E_{\frac{1}{2}}$ values of − 0·66 V and − 0·91 V. Bi interferes. A table of $E_{\frac{1}{2}}$ values (*vs.* S.C.E.) of 22 cations in the following media is given: (*a*) *M* NaOH plus 1 per cent. of Tiron; (*b*) *M* $(NH_4)_2CO_3$ plus 1 per cent. of Tiron; (*c*) as in (*b*) plus 0·02 *M* complexone III; (*d*) as in (*c*) with 0·05 *M* KCN added. G. GLASER

74. **Uranium determination in low-grade ores** L. Kosta (*Sloven. Acad. Sci. Arts, Ljubljana, Rep.*, 1953, **1**, 12–22).—The following methods of determining uranium in low-grade ores are described and discussed: an approx. radiometric method; a rapid accurate colorimetric method, based on the application of ammonium thioglycollate, that covers a wide range of concentrations down to 0·02 per cent.; and for lower concentrations (5×10^{-5} to 5×10^{-8} g), the rapid fluorimetric procedure for which the deviation does not exceed 7 per cent. For separating uranium from the accompanying elements, batch extraction with ethyl acetate in the presence of aluminium nitrate has proved very satisfactory in the last two methods. O. M. WHITTON

75. **Choice of supporting electrolytes for the polarographic determination of uranium in the presence of iron and copper.** M. V. Šusić (*Bull. Inst. Nuclear Sci., Belgrade*, 1954, **4**, 57–58).—The suitability of 0·5 *N* ascorbic acid, 0·5 *N* ammonium ascorbate, 0·5 *N* Na ascorbate, 0·2 *N* Na phthalate, 0·2 *N* ammonium phthalate, 0·1 *N* Na benzoate and 0·62 *N* ammonium benzoate as supporting electrolytes for the polarographic determination of U in low concn., alone or in presence of Fe and Cu, has been investigated. All give satisfactory results, but Fe and Cu occasionally interfere. L. F. TAYLOR

76. **Use of ion exchange for the determination of traces of impurities in uranium.** I. G. Draganić, Z. D. Draganić and Z. I. Dizdar (*Bull. Inst. Nuclear Sci., Belgrade*, 1954, **4**, 37–44).—The practicability of an ion-exchange method with the resin Amberlite IR–120 for the concentration, selective separation and quant. determination of traces of impurities in U has been investigated. U, Fe and Cu can be eluted with 0·5 *N* oxalic acid, Cd, Ni, Co and Mn with HCl, and rare earths with 5 per cent. ammonium citrate at pH 4·0. Except for Fe and Cu, the elements are determined polarographically, spectrometrically or colorimetrically. L. F. TAYLOR

77. **Paper partition chromatography of halogenated salts.** Y. Servigne (*Compt. Rend.*, 1954, **239** [3], 272–274).—The possibility of separating, identifying and determining quant. the different anions in mixtures of (*i*) chlorates, bromates and iodates, (*ii*) iodates and periodates, (*iii*) chlorites, chlorates, hypochlorites and chlorides has been examined by one-dimensional ascending paper partition chromatography of the Na and K salts. The

developing solvent is a (3 + 1, by vol.) mixture of *iso*propanol and H_2O, and the chromatogram is run for $\simeq$ 16 hr. For (*i*) the spots are revealed by spraying successively with a soln. of diphenylamine in conc. H_3PO_4 (for BrO_3' and IO_3') and in conc. HCl (for ClO_3'). R_F values are: $KClO_3$, 0·68; $KBrO_3$, 0·48; KIO_3, 0·20; and the min. amount detectable (of each ion or of one ion in presence of $\simeq$ 90 μg of the other two) is 3 to 3·6 μg per sample-drop (0·045 ml of a 0·6 per cent. soln.); periodate should be absent from the sample. Mixtures of (*ii*) cannot be separated satisfactorily because, although $Na_2H_3IO_6$ gives one clear spot (R_F 0·28), KIO_4 gives two overlapping spots (R_F, 0·21 and 0·28). For mixtures of (*iii*) the R_F values are: $NaClO_4$, 0·63 to 0·68; NaOCl and $NaClO_3$, 0·50 to 0·55, the spots (inseparable) are revealed with KI - starch soln.; NaCl, 0·46, the spot being revealed with 1 per cent. aq. $AgNO_3$ and 1 per cent. aq. K_2CrO_4. Owing to decomposition of chlorite and hypochlorite, the separation is not altogether reliable. W. J. BAKER

78. **Volumetric determination of small amounts of fluorides with ferric salts.** L. Sommer (*Chem. Listy*, 1953, **47** [6], 906–907).—To a soln. (10 to 15 ml) at pH 5 to 6 and containing < 10 mg of F′, add NaCl (2 g), 96 per cent. ethanol (10 ml) and 20 per cent. KCNS (5 drops). Titrate with 0·05 *N* $FeCl_3$ (acidity 0·01 *N* HCl) to faintly yellow coloration. Cover the surface of liquid with ether and complete the titration until the ethereal layer turns pink. G. GLASER

79. **Stabilised murexide indicator for the cerous chloride titration of fluorine.** G. Brunisholz (*Helv. Chim. Acta*, 1954, **37** [5], 1546–1547).—Murexide (200 mg) and $MgCl_2.6H_2O$ (1 g) are shaken vigorously with absolute ethanol (100 ml). The filtered solution is unchanged after 4 months if kept in brown flasks, even if these are opened frequently. The indicator is particularly suited to the titration of F, but can be used for the complexometric determination of Ca (titration in presence of Na), of Cu and Co in buffered ammoniacal solution (*p*H $\simeq$ 9), but not for Ni. H. WREN

80. **Mercurimetric determination of chloride.** J. F. Thomas (*J. Amer. Wat. Wks. Ass.*, 1954, **46** [3], 257–262).—The titration of chloride in waters and effluents with $Hg(NO_3)_2$, with diphenylcarbazone as indicator (Clarke, *Brit. Abstr. C*, 1950, 441), is more accurate and precise than Mohr's method because of the sharper end-point. The addition of bromophenol blue enables the pH to be adjusted to the optimum value (3·3) before the titration. A. WEBSTER

81. **Acidimetry of hypochlorites.** F. Provvedi (*Chim. e. Ind.*, 1954, **36** [7], 541–542).—The total alkali is determined by titration with 0·1 *N* hydrochloric acid directly after addition of hydrogen peroxide, and then the hypochlorite is determined by titration with oxalic acid (0·1 *N*) at the boil; the reaction is $2NaOCl + 2H_2C_2O_4 \longrightarrow Na_2C_2O_4 + 2CO_2 + Cl_2 + 2H_2O$. The difference between the two titres gives a figure that allows the amount of active chlorine to be calculated. R. C. MURRAY

82. **A volumetric determination for perchlorate.** D. C. Eagles (*Chem. & Ind.*, 1954, [33], 1002–1003).—The volumetric determination of perchlorates with titanous chloride as reducing agent and ferric alum in back-titration is modified by replacing the ferric alum with ceric ammonium sulphate and using the redox indicator diphenylaminesulphonic acid. The perchlorate samples are dissolved in conc. H_2SO_4, and, during the determination, an inert atmosphere is maintained by the presence of small pieces of solid CO_2 in the solutions. Standard 0·1 *N* ceric ammonium sulphate is run into the cool reduced solution until it is nearly colourless and then a 1 per cent. soln. of diphenylaminesulphonic acid indicator is added. The end-point is reached when a purple colour lasts at least 1 min. The method is suitable for rapid routine work, an inert atmosphere must be present over the titanous chloride at all times, and the blank values must be determined at frequent intervals. R. J. MAGEE

83. **Polarographic determination of iodine and iodides.** J. V. A. Novák (*Chem. Listy*, 1953, **47** [6], 903–904).—The method, based on the oxidation of I or I′ with NaOBr and on polarographic determination of IO_3', is applied to the analysis of common salt. Treat a soln. of the sample (2 g) in water (11 ml) with 1 ml of aq. NaOBr (from equal volumes of 5 *M* NaOH and saturated bromine water) and add 1 ml of a soln. prepared by mixing equal volumes of saturated Na_2SO_3 with 0·25 per cent. gelatin soln. Polarograph at 0·5 to 1·4 V. Compare the result with a standard prepared from the sample (2 g), water (10 ml) and KI (0·05 mg) in water (1 ml). G. GLASER

84. **The determination of periodate in the presence of carbohydrates and their oxidation products.** J. C. P. Schwarz (*Chem. & Ind.*, 1954, [33], 1000).—The uptake of periodate in the oxidation of carbohydrates is usually followed by reaction with KI in neutral solution, the iodine liberated being determined with standard arsenite soln. This method has been found unreliable when compounds containing activated hydrogen, *e.g.*, ethyl acetoacetate and ethyl α-ethylacetoacetate, occur as intermediates. In the presence of HI this error will not occur, and, as excess of HI is present in the "thiosulphate" method for the determination of periodate, this second method should be used when these intermediates occur. R. J. MAGEE

85. **Reaction of 2:2′-dipyridyl with iron in presence of organic matter.** M. Schnitzer and W. A. Delong (*Canad. J. Agric. Sci.*, 1954, **34** [3], 324–325).—Results obtained by the dipyridyl method for Fe content in presence of oxidisable organic matter are unreliable. P. S. ARUP

86. **Amperometric determination of ferricyanide with silver nitrate.** B. Khosla and H. C. Gaur (*Curr. Sci.*, 1954, **23** [7], 216–217).—The method described for determining low concn. of $K_3Fe(CN)_6$ involves amperometric titration with $AgNO_3$ and the manual polarographic arrangement (*cf. J. Indian Chem. Soc.*, 1953, **30**, 622), with a dropping-mercury electrode as cathode and an external saturated-calomel electrode as reference anode. L. F. TAYLOR

87. **Polarographic determination of nickel and cobalt in sintered carbides (hard metals).** K. Protiva (*Chem. Listy*, 1953, **47** [7], 1095–1097).—The polarographic determination of Co in sintered carbides has many advantages, especially in the presence of nickel, and takes less time than potentiometric titration with KCN. Heat the powdered sample (1 g) with conc. HNO_3 (30 ml) at 110° to 130° C during 30 min., dilute with water (30 ml), keep at the same temp. for 15 min., add conc. HCl (15 ml) and heat for a further 5 min. Samples containing Ti require heating at least for 30 min.

after dilution with water. Transfer the solution and sediment to a 100-ml calibrated flask, make up to the mark with water, shake and filter. Evaporate a 20-ml aliquot portion (50 ml for Co content < 2 per cent.) to dryness, dissolve the residue in dil. (1 + 1) HCl (5 ml), evaporate twice with the same vol. of HCl, finally dissolve in dil. (1 + 1) HCl (2·5 ml), dilute with water, add NH_4Cl (3 g), pyridine (3 ml), 1 per cent. gelatin soln. (5 ml), make up to 100 ml with water, filter, if necessary, and polarograph. HF can be used in place of HNO_3 to dissolve the sample of carbide.

G. GLASER

88. **Spectrophotometric studies of platinum - *p*-nitrosodimethylaniline complexes: colorimetric determination of trace amounts of platinum and palladium.** J. J. Kirkland (*Dissert. Abstr.*, 1954, **14** [5], 760).—The use of the colour reaction between Pt and dimethyl-*p*-nitrosoaniline as a sensitive colorimetric reagent for Pt has been investigated. Data on the structure of the coloured complexes has been collected, and a method reported for platinum determination has an accuracy range of 0·7 to 2·4 p.p.m. The similarity of the absorption curves of the Pd and Pt complexes and the difference in their reaction rates with dimethyl-*p*-nitrosoaniline makes possible an "additive adsorbency" colorimetric method to determine the two metals simultaneously without separation. An extraction procedure involving Na diethyldithiocarbamate is reported for selectively separating Pd and Pt from other interfering platinum-group metals, whereby, if combined with "additive absorbency" colorimetric determination, small amounts of Pd and Pt may be determined in the presence of other platinum-group metals. Small amounts of Pt (0·1 p.p.m., or 0·3 to 11 p.p.m. optimum) may be determined spectrophotometrically by making use of the 262-mμ absorbancy max. of $PtCl_6''$, but fairly complete separation is essential first, because of serious interference from most heavy metal chlorides.

L. F. TAYLOR

89. **The carbon effect in the spectrographic analysis of refractory materials.** R. Ricard (*Applied Spectroscopy*, 1954, **8** [2], 75–80).—The addition of graphite powder to samples of refractory powders results in the enhancement of lines of the less volatile elements, Al, Mg, Si and Ti, when the samples are excited in the crater of a carbon arc. Two factors which probably contribute to this effect are reduction of the refractory oxides by the graphite and the inhibition of globule formation in the crater.

B. S. COOPER

90. **Determination of oxygen-linked nitrogen in products of oxidation of coal [with nitric acid].** K. G. Beck (*Brennstoff-Chem.*, 1954, **35** [13–14], 212–213).—The oxidised product (1 g) is shaken in a separating funnel with 20 ml of zinc amalgam (500 pt. of Hg and 10 pt. of Zn) and 50 ml of 4 *N* HCl (*cf. Brit. Abstr. C*, 1950, 60). After separation of the Hg and cooling, the reduced amino groups are titrated with *N* $NaNO_2$, starch - KI paper being used as indicator. The oxidation products of a coal were separated by ether into soluble and insoluble parts in which, respectively, 70 to 80 and 35 to 40 per cent. of the N was reducible to $\cdot NH_2$. The remaining N is considered to be heterocylic, and includes that originally present in the coal.

A. R. PEARSON

See also Abstracts 8, 15, 29, 91, 129, 179, 188, 194, 204, 205, 213, 249.

3.—ORGANIC ANALYSIS

91. **Determination of isotopic carbon in organic compounds.** K. E. Wilzbach and W. Y. Sykes (*Science*, 1954, **120**, 494–496).—In the simplified procedure described, the sample is heated with CuO in a sealed tube and the CO_2 produced is isolated by fractional condensation *in vacuo*. Results by this method are reproducible to 1 per cent. and agree with values given by more elaborate combustion methods.

H. F. W. KIRKPATRICK

92. **Micro-determination of fluorine in organic compounds: amperometric methods.** B. H. Johnson, jun. (*Dissert. Abstr.*, 1954, **14** [5], 763–764).—Investigations are reported on the general application of an amperometric titration involving the reaction of F' with Ca^{++} in the presence of quaternary ammonium salts as supporting electrolytes. Br, N and S do not interfere, and the mean error is $\pm$ 0·5 per cent. for F. The F is separated by burning the material in O and trapping in 0·1 *M* KCl or KBr.

L. F. TAYLOR

93. **Rapid method for the determination of sulphur and chlorine in organic compounds.** O. Mikl and J. Pech (*Chem. Listy*, 1953, **47** [6], 904–906).—The previously described method for the determination of chlorine in organic compounds (Mikl and Pech, *Chem. Listy*, 1952, **46**, 382) has been applied to the determination of S, and S and Cl. A filter-paper containing $\simeq$ 100 mg of the substance is burnt in an oxygen-filled flask containing water (100 ml) and 3 per cent. neutral H_2O_2 (10 ml). After 1 hr., CO_2 is expelled by boiling for 2 min. If S is present, H_2SO_4 is titrated with NaOH. If S and Cl are present, total acidity is determined to methyl red, then the Cl content is found by titration with $Hg(NO_3)_2$ after acidifying with HNO_3.

G. GLASER

94. **Qualitative analysis of organic compounds by mass spectrometry.** J. H. Beynon (*Nature*, 1954, **174**, 735–737).—By examining the mass - charge ratios and the relative abundance of the various positive-ion species formed when the vapour of an unknown material is bombarded with electrons of $\simeq$ 50 eV, a qualitative analysis is possible. The method is based on empirical relationships deduced from a large number of mass spectra data. The mass measurement of the parent peak leads to the mol. wt. and the empirical formula of the compound, which is comparatively easy to identify from the above-mentioned rules if it is a hydrocarbon. When other elements are present, numerous atomic arrangements can be made for all but the lightest ion species. To overcome this difficulty the method is extended by introducing the technique of accurate mass measurement, based on the fact that atomic masses are not exactly integral. Reference samples are introduced, so as to give mass peaks of known composition as close as possible to the peaks being measured. With the identification of fragments of a mol., and a knowledge of its i.r. spectrum, the method can be extended to suggest a structural formula for a compound of unknown constitution. This has been successfully applied for compounds of mol. wt. < 200.

J. H. WATON

95. **Identification of organic compounds. III. Alkylthiuronium salts.** M. Jureček and M. Večeřa (*Coll. Czech. Chem. Comm.*, 1954, **19** [1], 77–84).—Various alkylthiuronium salts are investigated for the identification of alkyl and halogenoalkyl groups

attached to O or N. The unknown substance is heated with HI; the alkyl iodide formed is characterised by conversion to the thiuronium iodide and thence to its picrate (*cf. Chem. Listy*, 1951, **45**, 149 and 150) or another salt. 3:5-Dinitrobenzoates are now recommended on account of their lower solubility in alcohol. Other salts examined include styphnates, picrolonates, toluene-*p*-sulphonates, oxalates, nitrates and perchlorates. [This is a translation into German of a paper that appeared in *Chem. Listy*, 1952, **46**, 722.] D. R. GLASSON

96. **Remarks on the determination of primary and secondary alcohols by acetylation [in presence of] pyridine.** Y.-R. Naves and P. Ardizio (*Ann. Pharm. Franç.*, 1954, **12** [5], 357–361).—The method prescribed in the French Pharmacopoeia (1949) for the determination of primary and secondary alcohols in essential oils is criticised, in view of the inaccuracy of the volumetric measurements involved and the requirement of a back-titration. In the method proposed, a weighed amount of the oil, containing $\simeq$ 0·03 mole of alcohols, is placed in a 2·5 to 3·0-ml ampoule, and 0·6 ml of a mixture of 100 parts of acetic anhydride and 10 parts of pyridine (found to give least error in the acetylation of tertiary alcohols) is added, the exact amount being determined by weighing. The sealed ampoule and a blank are heated for 15 min. in boiling xylene. The ampoules are broken under 50 ml of water, and, after 3 min., are titrated with 0·5 *N* NaOH (phenolphthalein). Comparative results for oils of peppermint and rose are given for the two methods. E. J. H. BIRCH

97. **Paper chromatography of aldehydes and ketones as Girard derivatives.** R. B. Seligman, M. D. Edmonds, A. E. O'Keefe and L. A. Lee (*Chem. & Ind.*, 1954, [39], 1195).—A preliminary report is presented on the use of Girard's reagents for the isolation, separation and identification of pure carbonyl compounds from natural or synthetic mixtures, together with a list of R_F values of Girard T and P derivatives in butanol - ethanol - water. Substituted furfuraldehydes and benzaldehydes form solid derivatives with Girard's reagents, as do most of the aliphatic compounds investigated. Excellent separation of *o*-hydroxybenzaldehyde from the para compound and of 4-hydroxy-3-methoxybenzaldehyde from the corresponding ethoxy compound is attained. The aliphatic, aromatic and heterocyclic compounds differ in their u.v. spectra. J. M. JACOBS

98. **Spectrophotometric method for the estimation of ammonium acetate with sensitised Schiff's reagent.** S. N. Bhattacharya and A. Ghose (*Anal. Chim. Acta*, 1954, **11** [3], 244–248).—When a soln. of ammonium acetate is added to Schiff's aldehyde reagent, the colour of the fuchsin is restored. This reaction is used for the estimation of ammonium acetate. The sensitised Schiff's reagent of Tobie (*Brit. Abstr. A II*, 1942, 279) is used. Sensitivity towards ammonium acetate and the concn. in which it can be determined depend upon the concn. of SO_2 in the reagent. The sensitivity and range can also be altered by adding acetone. The time interval recommended for colour development is 15 min. The light absorption is measured at 535 mμ and standard graphs are prepared. Max. error for 6 to 24-mg amounts of ammonium acetate is 1·4 per cent. W. C. JOHNSON

99. **Organic qualitative analysis. IV. Identification of carboxylic acids with S-(*p*-nitrobenzyl)- and S-(2:4-dinitrobenzyl)-thiuronium chloride.** Tsutomu Momose and Hiroko Tanaka (*Pharm. Bull., Japan*, 1954, **2** [2], 152–154).—Details are given for preparing S-(*p*-nitrobenzyl)thiuronium chloride, m.p. 223° C, and S-(2:4-dinitrobenzyl)thiuronium chloride, m.p. 202° C, by refluxing thiourea with *p*-nitro- or 2:4-dinitrobenzyl chloride in ethanol. These products are stable in air and can be used for identifying and separating carboxylic, sulphonic and amino-acids. The general method of preparing thiuronium salts of these acids by mixing the reagent and their sodium salts is described. Melting points and crystal forms of the salts are tabulated. Carboxylic acids are easily regenerated from these thiuronium salts by acidifying with HCl. O. M. WHITTON

100. **High-frequency titration. XI. Non-aqueous titration by high-frequency method. Titration of salts of organic acids.** Morizo Ishidate and Masaichiro Masui (*Pharm. Bull., Japan*, 1954, **2** [1], 50–53).—Na oxalate, benzoate, salicylate and citrate, Pb acetate, Ca lactate and gluconate, $KHCO_3$ and K Na tartrate were determined by high-frequency titration in non-aqueous solutions, in a mixture of methanol and benzene, and, if necessary, acetic acid and ethylene glycol, with perchloric acid or Na acetate in acetic acid. Na salts were generally titrated directly with perchloric acid with an accuracy of 0·2 to 0·3 per cent., K salts were back-titrated with an accuracy of about 0·4 per cent., and Ca and Pb salts with an accuracy of 0·4 to 0·7 per cent. O. M. WHITTON

101. **Polarographic determination of carbon oxysulphide.** V. Šedivec and V. Vašák (*Chem. Listy*, 1954, **48** [1], 19–26).—The method is based on the reaction of COS with an ethanolic soln. of diethylamine; the diethylmonothiocarbamic acid formed is polarographically active and gives an anodic wave at $-$ 0·32 V. The dependence of the wave height on the reservoir height, on the temp. and on the concn. is discussed. The method enables COS to be determined in the presence of H_2S, CS_2 and mercaptans, and has been applied to the analyses of illuminating and generator gases. *Procedure*—Absorb the gas in a 1 per cent. soln. of diethylamine in ethanol. To 10 ml of this soln., add 2 *M* $LiNO_3$ (1 ml), displace O with N, and polarograph, using the anodic - cathodic polarisation and the back course from the eighth winding of the potentiometric wire. G. GLASER

102. **Polarographic study of dithiocarbamic acids.** R. Zahradník and L. Jenšovský (*Chem. Listy*, 1954, **48** [1], 11–18).—The polarographic behaviour of the salts of N-dithiocarboxyglycine (**I**) and N-dithiocarboxyalanine (**II**) was studied. Their diffusion-controlled anodic wave at pH 11 was found to be most suitable for analytical purposes, its height being a linear function of concn. in the range 10^{-3} to 10^{-5} *M*. The analysis of the polarographic curve supports the reaction scheme suggested by Kolthoff and Barnum (*J. Amer. Chem. Soc.*, 1940, **62**, 3061) for the electrolytic process as RSH + Hg $\rightleftharpoons$ HgSR + H^+ + e. The observed dependences of the height of the anodic wave of **I** at pH 11 on the square root of the reservoir height and on the temp., as well as the agreement of the observed temp. coeff. (1·73 per cent.) with the theoretical (1·63 per cent.), are in accordance with the criteria for diffusion-controlled phenomena. The formation of **I** and **II** from CS_2

and the amino-acid in alkaline media was followed polarographically, with a view to using the reaction for the determination of these acids. Further, the decomposition of the dithiocarbamic acids at pH 1·70, 2·58 and 3·47 was studied and found to be of the first order; the activation energies and entropies of the reaction were calculated. G. GLASER

103. **Organic qualitative analysis. V. Paper chromatography of aromatic hydrocarbons and ethers.** Tsutomu Momose, Yosuke Ohkura and Hiroko Tanaka (*Pharm. Bull., Japan*, 1954, **2** [2], 154–156).—The paper chromatography of derivatives of aromatic hydrocarbons and ethers is reported. First, the aromatic hydrocarbon or ether was condensed with succinic anhydride by the Friedel - Crafts method. The Na β-aroylpropionate obtained was spotted on to filter-paper and developed with butanol - NH_3. The chromatogram was dried and then coloured by spraying with sodium nitroprusside and NaOH. The results obtained with naphthalene, anisole and *p*-dichlorobenzene are given and discussed. Secondly, aromatic hydrocarbon or ether was converted to the Ba phenylsulphonate by sulphonating with chlorosulphonic acid and heating with H_2O and $BaCO_3$. The Ba salt was developed with butanol - NH_3. Methyl red indicator was used for detecting the paper chromatograms. R_F values are tabulated of the β-aroylpropionic acids and phenylsulphonic acids derived from benzene, toluene, *m*-xylene, naphthalene, tetralin, anisole, veratrole, *p*-dimethoxybenzene, 2:5-dimethoxytoluene and chlorobenzene, and the aroylpropionic acids derived from *m*-dimethoxybenzene and *p*-dichlorobenzene. O. M. WHITTON

104. **The spectrophotometric response of diphenyl and anthracene.** J. M. Vandenbelt and C. Henrich (*Analyst*, 1954, **79**, 586–588).—Davenport (*Brit. Abstr. C*, 1953, 512) has reported that solutions of diphenyl show far from linear response at high optical densities when the Beckman spectrophotometer is used. It is likely that this departure is principally due to characteristics of the instrument. The optical densities of solutions of diphenyl in *cyclo*hexane in the concn. range used by Davenport were measured on Beckman and Cary spectrophotometers concurrently, the latter being designed to record optical densities up to 3·4. The results with the Beckman instrument were similar to those reported above (*loc. cit.*); the response with the Cary instrument was linear, indicating no departure from Beer's law. The shape of the graph with the Beckman instrument was different from that characteristic of a fluorescent material. Graphs with anthracene in *cyclo*hexane, in contrast with those of diphenyl, are not linear in the Cary instrument, owing presumably to the effect of the intense fluorescent band at 382 mμ. Deviations from Beer's law can thus be due to characteristics of the instrument or the specific properties of compounds, and combinations of instrument and compound should be examined carefully for the cause of such discrepancies. A. O. JONES

105. **Chlorobenzene.** British Standards Institution (B.S. 2533: 1954, 7 pp.).—This specification for chlorobenzene prescribes methods for determination of density, distillation range, residue on evaporation, acidity (100 ml must not change the colour of bromophenol blue in 50 ml of neutralised water), and free chlorine (no yellow colour to be developed with *o*-tolidine hydrochloride solution).
E. J. H. BIRCH

106. **A new micro method for the analysis of phenols.** B. T. Commins and R. L. Cooper (*Chem. & Ind.*, 1954, [36], 1110).—Cooper's method for the analysis of polynuclear aromatic compounds on the microgram scale (*cf. Brit. Abstr. C*, 1953, 470) has been extended to include simple phenols. Procedure involves (*i*) methylation of the phenol-containing mixture, (*ii*) chromatography of the resulting ethers on an activated-alumina column with *cyclo*hexane as solvent and (*iii*) spectroscopic examination of the column eluates. The method is more rapid than paper chromatography and identification is more precise, except that phenols containing methoxy groups cannot be distinguished from those with hydroxy groups only. L. F. TAYLOR

107. **Dibutyl sebacate.** British Standards Institution (B.S. 2535: 1954, 9 pp.).—This specification for dibutyl sebacate, which has clauses on colour, colour stability and viscosity, included to meet the requirements of the plastics industry, prescribes a colour of < 3 yellow units and 1 red unit on the Lovibond scale (6-in. cell), and after heating to 180° C (15 min.) < 4·5 yellow units and 1·5 red units, limits of *d*, n_D, ash, water content (Karl Fischer), acidity (< 0·04 per cent. free acid), volatile matter (< 1·0 per cent. in 1 hr. and < 1·5 per cent. in 2 hr. at 130° C), η and ester content (by saponification with *N* ethanolic KOH).
E. J. H. BIRCH

108. **Di-2-ethylhexyl sebacate.** British Standards Institution (B.S. 2536: 1954, 9 pp.).—This specification for di-2-ethylhexyl sebacate, which has clauses on colour and colour stability included to meet the requirements of the plastics industry, prescribes a colour of < 4·0 yellow units and 1·5 red units on the Lovibond scale (6-in. cell), and after heating to 180° C (15 min.) < 6·0 yellow units and 2·5 red units, limits of *d*, n_D, ash, water content (Karl Fischer), acidity (< 0·04 per cent. free acid), volatile matter (< 0·5 per cent. in 1 hr. and < 0·75 per cent. in 2 hr. at 130° C), η and ester content (by saponification with *N* ethanolic KOH).
E. J. H. BIRCH

109. **Detection of NN-dimethylaniline in esterification reactions.** K. Kukla (*Amer. Perfum.*, 1954, **64** [2], 101–102).—In the esterification of sensitive alcohols, *e.g.*, tertiary and terpene alcohols, an acid chloride and a tertiary base, *viz.*, NN-dimethylaniline (**I**), are frequently used in place of an acid anhydride if the latter is unsuitable. Quality considerations demand complete freeing of the final product from **I**, either by repeated washing with aq. mineral acid or by careful fractionation. A quick and reliable test is based on the ready oxidation of **I** to tetramethylbenzidine, which in acid solution forms a water-soluble orange-yellow compound. In purification by acid washing, 15-ml portions of successive wash layers are treated with 1 to 3 drops of saturated aq. $KMnO_4$; if **I** is present the colour develops within 15 sec. In fractionation purification, 10 ml of the fraction are shaken with 15 ml of 5 per cent. H_2SO_4 and the separated aqueous acid is then treated with the $KMnO_4$ solution as before.
G. HELMS

110. **Terpenes. LVI. Paper chromatography of azulenes.** O. Knessl and A. Vlastiborová (*Chem. Listy*, 1954, **48** [2], 212–216).—The separation and identification of a number of azulenes by paper chromatography is described, paraffin oil being used as the stationary phase and aq. H_3PO_4 as the ascending mobile phase. The spots are detected by washing

the chromatogram with water or, quantitatively, by exposing it to NH_3 gas. The effect of the degree of impregnation of the filter-paper (Whatman No. 1) with the oil, of the concn. of the acid and of the amounts of individual azulenes chromatographed on their separation is discussed, and a table of R_F values corresponding to H_3PO_4 concn. from 35 to 70 per cent. is given. G. GLASER

111. **Chromatographic semimicro-analysis of gases. IV and V. Separation and analysis of gaseous hydrocarbons.** J. Janák (*Coll. Czech. Chem. Comm.*, 1954, **19** [4], 700–711).—The chromatographic separation and analysis of gaseous paraffins, C_2- and C_3- unsaturated hydrocarbons and *cyclo*propane are described. The apparatus described previously (*Anal. Abstr.*, 1954, **1**, 2991) is suitably modified for chromatography of these gases. Reproducibility and accuracy of analytical results are determined by analysis of known gas mixtures with various adsorbents. Gases with the same number of C atoms, *e.g.*, C_2H_2, C_2H_4 and C_2H_6, show varying behaviour towards different adsorbents; the elution vol. for active C increases with the degree of saturation of the molecule but decreases for silica gel. Sorption on active C is largely determined by mol. size, but on silica gel it depends mainly on the energy content of the mol. Thus C_2H_2 and C_3H_8 are separable on active C, but not on silica gel, whilst propyne and C_2H_2 are separable on both adsorbents. Analytical results are compared with those obtained by other methods for natural gas, coke-oven gas, cracked-oil gases, illuminating gas, water gas, carbonising gas, Autogas and synthesic gas (methanol). [This is a translation into German of two papers that appeared in *Chem. Listy.*, 1953, **47**, 1184 and 1190.] D. R. GLASSON

112. **Chromatographic semimicro-analysis of gases. VIII. Separation and analysis of some halogenated hydrocarbons.** J. Janák and M. Rusek (*Chem. Listy*, 1954, **48** [2], 207–211).—The chromatographic separation and analysis of mixtures of halogenated hydrocarbons is described. The following R_F values were obtained on silica gel with the passage of the eluting gas (CO_2) at the rate of 0·7 ml per sec.: CH_3Cl, 0·0039; CH_3Br, 0·0020; CH_2:CHCl, 0·0024; CF_2:CFCl, 0·0099 at 20° C; CH_3Cl, 0·159; CH_3Br, 0·120; C_2H_5Cl, 0·104; CF_2:CFCl, 0·214 at 85° C; and CH_2:CHCl, 0·110 at 65° to 70° C. In view of the considerable influence of temp. on the adsorption characteristics of this group of gases, better separation is achieved at 20° than at 85° C. Analytical applications of the method are limited owing to partial hydrolysis of the halides during their contact with the KOH in the azotometer, resulting in a decrease of the measured vol. The reactivity increases in the order CF_2:CFCl < CH_2:CHCl < CH_3Cl < C_2H_5Cl. The determination of CH_3Br is impossible. G. GLASER

113. **The determination of polycyclic hydrocarbons in town air.** R. L. Cooper (*Analyst*, 1954, **79**, 573–579).—A method for determining polycyclic aromatic hydrocarbons in town air is described. Suspended matter in a measured vol. of air is collected daily by aspiration through filter-paper, the wt. of deposit being ascertained by means of a calibrated reflectometer. The papers are extracted with acetone, the extracted material is transferred into *cyclo*hexane soln., which is then poured on to an alumina column, and the chromatogram is developed with *cyclo*hexane. During development the eluates are examined spectrophotometrically at the peak wavelengths corresponding to individual hydrocarbons and, after suitable combinations of the eluate fractions, the hydrocarbons are determined spectrophotometrically. The results are considered in relation to the recent increase in lung cancer, as the carcinogen 3:4-benzopyrene is present in atmospheric smoke. A. O. JONES

114. **Analysis of the unsaturated hydrocarbons in fuel gases by infra-red spectroscopy. I. Analysis of a town's gas.** J. H. Towler and B. H. Holland (*J. Appl. Chem.*, 1954, **4** [9], 517–524).—A method proposed for analysis of the unsaturated hydrocarbon content of carbonisation gases and carburetted water gas involves (*i*) removal of condensable compounds by refrigeration at − 80° C, (*ii*) separation of the remaining unsaturated hydrocarbons as bromine addition compounds, (*iii*) fractionation of the 2 groups of compounds and (*iv*) analysis of the fractions by i.r. spectroscopy. Synthetic mixtures similar in composition to the fractions obtained in a town gas have been analysed satisfactorily, and application of the method to a town gas resulted in resolution of the unsaturated hydrocarbons into 37 individual compounds. Thus the composition of the compounds in the original town gas can be calculated, provided that the vol. of gas giving rise to known vol. of condensable and bromine compounds is known. L. F. TAYLOR

115. **Review of modern methods of chemical analysis [of petroleum products].** G. Chatelus (*Rev. Inst. Franç. Pétrole*, 1954, **9** [6], 308–320; [7], 403–411).—Methods applicable to petroleum products are reviewed briefly. They include determination of elements (N, O, S, halogens), water peroxides, and types of hydrocarbon, and analysis of gases (O_2, C_2N_2, CO) and mineral constituents (Fe, Ni, V, Cu). A bibliography contains 301 references to recent literature (mainly 1948–1953). A. R. PEARSON

116. **A test for mercaptans in gasoline.** G. E. Mapstone (*Chem. & Ind.*, 1954, [36], 1113–1114).—A modification of Rheinboldt's test for determination of mercaptans in gasoline involves shaking 5 ml of the sample with 1 ml of glacial acetic acid and adding a few $NaNO_2$ crystals. The method, when applied to butane-1-thiol in shale gasoline containing approx. 50 per cent. of olefins, is less sensitive than other tests, the colour developing slowly (1½ to 2 hr.). The test is severely hampered if the gasoline has been dyed or is discoloured; alcohol addition inhibits colour formation. L. F. TAYLOR

117. **Determination of different types of hydrocarbon [in motor spirit] by adsorption with fluorescent indication.** A. Crozier (*Rev. Inst. Franç. Pétrole*, 1954, **9** [7], 397–402).—The method consists in pouring ≃ 0·8 ml of the sample, to which a trace of a special fluorescent indicator has been added, on to a column of a silica gel (100–200 mesh) and displacing it downwards by ethanol, to which pressure can be applied, into a measuring tube of diameter 1·5 to 2 mm, where it forms a column. The first section of the column contains saturated hydrocarbons, the second, separated by a yellow fluorescent zone under mercury light, contains olefins, and the third, which fluoresces blue with a brown ring at its junction with the ethanol, contains aromatics. The lengths of the three sections are proportional to the vol. per cent. in the original petrol. The results are reliable within ± 1 per cent., tending to give a high value for aromatics and a

low value for olefins when compared with a synthetic mixture. The percentage of olefin is lower than that calculated from the bromine value. See also *Anal. Abstr.*, 1954, **1**, 984.

A. R. PEARSON

118. **Behaviour of bituminous substances in ultra-violet light.** H. Schmidt (*Erdöl u. Kohle*, 1954, **7** [7], 428–433).—A method of investigating bituminous substances by adsorption on filter-paper from decane solution and examining the product in filtered u.v. light is described. The characteristic patterns given by various bituminous substances are described. Mixtures can be dealt with, but the work is made considerably easier if known mixtures are available for comparison. A. J. MEE

119. **Identification of textile and related fibres.** J. E. Ford and W. J. Roff (*J. Text. Inst.*, 1954, **45** [8], 580–623).—This paper was first published as a Shirley Institute Memoir (*Anal. Abstr.*, 1954, **1**, 1901). L. VALENTINE

120. **Use of adsorption columns in the analysis of emulsions stabilised with non-ionic detergents.** T. Green, R. P. Harker and F. O. Howitt (*Nature*, 1954, **174**, 659).—Lanolin - "Lissapol N" emulsions, containing up to 1 per cent. of sodium chloride, have been separated on columns of "Bio-Deminrolit" (a mixture of Zeo-Karb 225 and De-Acidite FF). Quantitative yields of "Lissapol N" were obtained by elution with 50 per cent. aqueous ethanol, but when animal charcoal was added as an additional adsorbent the "Lissapol N" was too strongly adsorbed for solvent elution. Emulsions of "Lissapol N" with (*i*) a vegetable oil and (*ii*) a mineral oil have been analysed in this way, and the method has been extended to the analysis of a "Lissapol N" - soap - lanolin emulsion. D. LIFF

121. **Determination of hypericin in hypericum oil.** V. Hagenström (*Dtsch. ApothZtg.*, 1954, **94** [42], 1020–1022).—A photometric method is described for the determination of hypericin in hypericum oil; the oil contains $\not<$ 2 mg of hypericin per 100 ml, but a good sample may contain 3 mg. Alkannin can be identified by its light-absorption characteristics.

S. C. JOLLY

122. **The manner of reaction and the identification of polyvinyl and polyacrylic compounds.** H. Rath and L. Heiss (*Kunststoffe*, 1954, **44**, 341–347).—Chemical methods of identifying polyvinyl compounds and polyacrylates, and polymethacrylates in very small samples of polymers and co-polymers are described. The sample is dissolved in tetrahydrofuran, and passed through Al_2O_3 to remove any dye. Polyvinyl chloride, acetate and alcohol can be identified by the coloured polyenes formed when a stain on filter-paper is treated with Na ethoxide, Na butoxide, sodamide or thionyl chloride. Polyvinyl ethers react in solution with phenyllithium to give coloured polyenes. Polacrylates and polymethacrylates react in solution with phenyllithium to give polyvinyl-$\beta\beta$-diphenylcarbinols, which lose H_2O under the action of acetyl chloride to give the corresponding diphenylvinyls. Both these give characteristic colours with H_2SO_4, $ZnCl_2$, $SOCl_2$, etc. The alcohol component in the ethers and esters can be converted to the 3:5-dinitrobenzoate and identified by its m.p. The acetic acid from polyvinyl acetate can be isolated and titrated.

A. B. DENSHAM

123. **Synthetic resin (phenolic) moulding materials.** British Standards Institution (B.S. 771:1954, 40 pp.).—The requirements for various phenolic resin moulding materials (including high-dielectric, shock-resistant, heat-resistant and electrical low-loss types) are specified together with the prescribed tests for determining cross-breaking strength, impact strength, surface resistivity after immersion in water, vol. resistivity, heat resistance, power factor and permittivity, tensile strength, water absorption, plastic yield, dielectric strength (minute value at 90° C) and percentage of matter soluble in acetone. Test methods are also indicated for the determination of the apparent powder density and bulk factor, time of flow by the cup flow test, shrinkage, density, crushing strength, shear strength and elastic modulus in tension. J. M. JACOBS

124. **The Liebermann, Storch and Morawsky reaction.** M. Nortz, P. Dupuy and H. Rabaté (*Peint.Pigm.-Vern.*, 1954, **30** [7], 568–571).—The mechanism of the reaction, which is used as a test for rosin, has not been established, as the compounds giving the colours cannot be isolated. Colours produced by a wide range of oils and resins in the test are tabulated, but caution is required in the interpretation of the results.

L. A. O'NEILL

125. **Checking hiding power of paints in the control laboratory.** E. J. Dunn, jun. (*Paint Ind. Mag.*, 1954, **69** [6], 17–18).—Charts are constructed showing percentage reflections over black and white substrates against the reciprocal of film thickness for each standard paint. Production batches should then be checked, preferably at 2 film thicknesses, with reference to the charts. L. A. O'NEILL

126. **N.P.I.R.I. standard method for tinting strength.** W. C. Walkers (*Amer. Ink Maker*, 1954, **32** [2], 25–27, 63).—The National Printing Ink Research Institute of the U.S.A. has drawn up a series of standard test methods specially designed to test the suitability of pigments for use in printing ink. Method II-Ec-3, "Relative Tinting Strength of Pigment Pastes," is reproduced in full. The test is made in the normal way with pastes of the pigments in lithographic varnish mixed by grinding with a steel spatula. The standard pastes for mixing with pigments of different colours are: for testing white pigments, optionally either dark blue (based on 42 pt. of ultramarine plus 18 pt. of pptd. $CaCO_3$) or black (4 pt. of lamp black plus 43 pt. of pptd. $CaCO_3$); for yellow pigments, a light blue (0·150 pt. of flushed green-shade phthalocyanine blue plus 100 pt. of ZnO paste or ZnO paste alone); for all other colours and black, ZnO paste. Suitable proportions for mixing different pigments with these standard pastes are suggested.

D. R. DUNCAN

127. **Fluorescence and absorption spectroscopy applied in studies of certain laboratory products, isolated and purified by chromatographic and electrophoretic analyses.** M. O'L. Crowe and A. Walker (*Applied Spectroscopy*, 1954, **8** [2], 57–66).—The spectroscopic work carried out in the laboratories of the New York State Department of Health is described. Particular attention has been paid to the fluorescence spectra and absorption spectra of certain pigments of the porphyrin and pterin groups, *viz.*, coproporphyrin III, erythropterin and xanthopterin. B. S. COOPER

See also Abstracts 59, 65, 189, 194.

4.—BIOCHEMISTRY

INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

128. **The estimation of potassium in blood serum by the method of Kramer and Tisdall.** H. Chomse and I. Arend (*Hoppe-Seyl. Z.*, 1954, **297** [1], 45–46).—The value obtained depends on the concn. of K in the soln. to be estimated. The dilution brought about by the removal of proteins may lead to faulty results. If the volume used is reduced to 1 ml before precipitation, accurate results are obtained. A centrifuge tube marked at 1 ml is closed with a two-holed rubber stopper and dipped into a water-bath at 60° C while a stream of air is drawn through. The volume is reduced within a few min. and precipitation can then be satisfactorily carried out. F. POWELL

129. **A rapid method for the simultaneous determination of calcium and sodium in bone.** G. C. H. Bauer (*Acta Physiol. Scand.*, 1954, **31** [4], 351–358).—Samples (100 to 160 mg) of the fat-free dry powdered bone are shaken with 30 per cent. aq. trichloroacetic acid (3 ml) for 4 hr. Known amounts of $Na^{\cdot}$ and $Ca^{\cdot\cdot}$ are added to some of the samples before treatment with the acid, and the solutions are diluted to 100 ml before determination. Standard solutions are prepared from $Ca_3(PO_4)_2$ and NaCl in 30 per cent. aq. trichloroacetic acid; these contain approx. the same amounts of $Ca^{\cdot\cdot}$, $Na^{\cdot}$, $K^{\cdot}$, $Mg^{\cdot\cdot}$ and $PO_4^{\cdot\cdot\cdot}$ as those expected in the samples. The Ca emission of the samples is then determined by means of a spectrophotometer with a flame photometer attachment; the emission of two standards, one containing more Ca and one less than the sample, is also determined. Standards are then selected that have the same Ca emission as the sample, but which contain various amounts of Na. The Na emission of the sample is then determined with two of the standards, one containing more Na and one less than the sample. From the values so obtained, the amounts of $Ca^{\cdot\cdot}$ and Na, in the sample are calculated. J. N. ASHLEY

130. **Tests for alcohol in breath and blood.** J. M. Berkebile (*J. Chem. Educ.*, 1954, **31** [7], 380–382).—Law enforcement officers in the U.S. use the Intoximeter to determine the alcohol content in the breath and hence in the blood. The Intoximeter is a simple instrument comprising a balloon connected by a U-leg to (*a*) a tube containing a known amount of acidified $KMnO_4$ soln. and (*b*) tubes in series containing (*i*) pure $Mg(ClO_4)_2$ and (*ii*) Ascarite. A breath sample is collected in the balloon and first permitted to pass through the $KMnO_4$ soln.; the time taken to decolorise the soln. gives an immediate indication of the state of intoxication. For a permanent record, the remainder of the breath sample is passed through the $MgClO_4$, which absorbs the alcohol, and then through the Ascarite, which absorbs the CO_2; the two reagents can be subsequently analysed at leisure. G. HELMS

131. **Alcohol in post-mortem specimens. II. Comparative determinations by Widmark's and Zeisel - Fanto's methods and by the A.D.H. method.** R. Bonnichsen, F. Halstrøm, K. O. Møller and H. Theorell (*Acta Pharmacol. Tox., Kbh.*, 1954, **10** [2], 101–112).—From comparative results on post-mortem samples it is concluded that Widmark's method is unsatisfactory if more than 48 hours have elapsed between death and autopsy. Zeisel - Fanto's method, which is fully detailed, and the enzymatic A.D.H. method described by Bonnichsen *et al.* (*Scand. J. Clin. Lab. Invest.*, 1951, **3**, 58 and *Acta Pharmacol. Tox., Kbh.*, in the press), are found to give almost identical results even on putrified materials. W. H. C. SHAW

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133. **Anthrone reaction in estimation of carbohydrates.** F. Grande, A. Utrera and J. C. de Oya (*Bull. Inst. Med. Res., Univ. Madrid*, 1953, **6** [2], 67–81).—This is a translation into English of the paper abstracted in *Anal. Abstr.*, 1954, **1**, 1321. W. H. C. SHAW

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135. **Use of alpha diketones for analysis of urea.** R. C. Dickenman, B. Crafts and B. Zak (*Amer. J. Clin. Path.*, 1954, **24** [8], 981–987).—Two similar procedures for the chemical determination of serum urea are presented; they involve the use of simply prepared, stable and sensitive colour reagents. The postulated mechanism of reaction, along with spectral curves, calibration curves and time-of-heating curves are included as well as tables which indicate that suitable precision and accuracy can be attained. The α-diketones diacetyl and acetylbenzoyl are simpler to use than the corresponding

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low value for olefins when compared with a synthetic mixture. The percentage of olefin is lower than that calculated from the bromine value. See also *Anal. Abstr.*, 1954, **1**, 984.

A. R. PEARSON

118. **Behaviour of bituminous substances in ultra-violet light.** H. Schmidt (*Erdöl u. Kohle*, 1954, **7** [7], 428–433).—A method of investigating bituminous substances by adsorption on filter-paper from decane solution and examining the product in filtered u.v. light is described. The characteristic patterns given by various bituminous substances are described. Mixtures can be dealt with, but the work is made considerably easier if known mixtures are available for comparison. A. J. MEE

119. **Identification of textile and related fibres.** J. E. Ford and W. J. Roff (*J. Text. Inst.*, 1954, **45** [8], 580–623).—This paper was first published as a Shirley Institute Memoir (*Anal. Abstr.*, 1954, **1**, 1901). L. VALENTINE

120. **Use of adsorption columns in the analysis of emulsions stabilised with non-ionic detergents.** T. Green, R. P. Harker and F. O. Howitt (*Nature*, 1954, **174**, 659).—Lanolin - "Lissapol N" emulsions, containing up to 1 per cent. of sodium chloride, have been separated on columns of "Bio-Deminrolit" (a mixture of Zeo-Karb 225 and De-Acidite FF). Quantitative yields of "Lissapol N" were obtained by elution with 50 per cent. aqueous ethanol, but when animal charcoal was added as an additional adsorbent the "Lissapol N" was too strongly adsorbed for solvent elution. Emulsions of "Lissapol N" with (*i*) a vegetable oil and (*ii*) a mineral oil have been analysed in this way, and the method has been extended to the analysis of a "Lissapol N" - soap - lanolin emulsion. D. LIFF

121. **Determination of hypericin in hypericum oil.** V. Hagenström (*Dtsch. ApothZtg.*, 1954, **94** [42], 1020–1022).—A photometric method is described for the determination of hypericin in hypericum oil; the oil contains $\leqslant$ 2 mg of hypericin per 100 ml, but a good sample may contain 3 mg. Alkannin can be identified by its light-absorption characteristics. S. C. JOLLY

122. **The manner of reaction and the identification of polyvinyl and polyacrylic compounds.** H. Rath and L. Heiss (*Kunststoffe*, 1954, **44**, 341–347).—Chemical methods of identifying polyvinyl compounds and polyacrylates, and polymethacrylates in very small samples of polymers and co-polymers are described. The sample is dissolved in tetrahydrofuran, and passed through Al_2O_3 to remove any dye. Polyvinyl chloride, acetate and alcohol can be identified by the coloured polyenes formed when a stain on filter-paper is treated with Na ethoxide, Na butoxide, sodamide or thionyl chloride. Polyvinyl ethers react in solution with phenyllithium to give coloured polyenes. Polacrylates and polymethacrylates react in solution with phenyllithium to give polyvinyl-$\beta\beta$-diphenylcarbinols, which lose H_2O under the action of acetyl chloride to give the corresponding diphenylvinyls. Both these give characteristic colours with H_2SO_4, $ZnCl_2$, $SOCl_2$, etc. The alcohol component in the ethers and esters can be converted to the 3:5-dinitrobenzoate and identified by its m.p. The acetic acid from polyvinyl acetate can be isolated and titrated.

A. B. DENSHAM

123. **Synthetic resin (phenolic) moulding materials.** British Standards Institution (B.S. 771:1954, 40 pp.).—The requirements for various phenolic resin moulding materials (including high-dielectric, shock-resistant, heat-resistant and electrical low-loss types) are specified together with the prescribed tests for determining cross-breaking strength, impact strength, surface resistivity after immersion in water, vol. resistivity, heat resistance, power factor and permittivity, tensile strength, water absorption, plastic yield, dielectric strength (minute value at 90° C) and percentage of matter soluble in acetone. Test methods are also indicated for the determination of the apparent powder density and bulk factor, time of flow by the cup flow test, shrinkage, density, crushing strength, shear strength and elastic modulus in tension. J. M. JACOBS

124. **The Liebermann, Storch and Morawsky reaction.** M. Nortz, P. Dupuy and H. Rabaté (*Peint.Pigm.-Vern.*, 1954, **30** [7], 568–571).—The mechanism of the reaction, which is used as a test for rosin, has not been established, as the compounds giving the colours cannot be isolated. Colours produced by a wide range of oils and resins in the test are tabulated, but caution is required in the interpretation of the results.

L. A. O'NEILL

125. **Checking hiding power of paints in the control laboratory.** E. J. Dunn, jun. (*Paint Ind. Mag.*, 1954, **69** [6], 17–18).—Charts are constructed showing percentage reflections over black and white substrates against the reciprocal of film thickness for each standard paint. Production batches should then be checked, preferably at 2 film thicknesses, with reference to the charts. L. A. O'NEILL

126. **N.P.I.R.I. standard method for tinting strength.** W. C. Walkers (*Amer. Ink Maker*, 1954, **32** [2], 25–27, 63).—The National Printing Ink Research Institute of the U.S.A. has drawn up a series of standard test methods specially designed to test the suitability of pigments for use in printing ink. Method II-Ec-3, "Relative Tinting Strength of Pigment Pastes," is reproduced in full. The test is made in the normal way with pastes of the pigments in lithographic varnish mixed by grinding with a steel spatula. The standard pastes for mixing with pigments of different colours are: for testing white pigments, optionally either dark blue (based on 42 pt. of ultramarine plus 18 pt. of pptd. $CaCO_3$) or black (4 pt. of lamp black plus 43 pt. of pptd. $CaCO_3$); for yellow pigments, a light blue (0·150 pt. of flushed green-shade phthalocyanine blue plus 100 pt. of ZnO paste or ZnO paste alone); for all other colours and black, ZnO paste. Suitable proportions for mixing different pigments with these standard pastes are suggested.

D. R. DUNCAN

127. **Fluorescence and absorption spectroscopy applied in studies of certain laboratory products, isolated and purified by chromatographic and electrophoretic analyses.** M. O'L. Crowe and A. Walker (*Applied Spectroscopy*, 1954, **8** [2], 57–66).—The spectroscopic work carried out in the laboratories of the New York State Department of Health is described. Particular attention has been paid to the fluorescence spectra and absorption spectra of certain pigments of the porphyrin and pterin groups, *viz.*, coproporphyrin III, erythropterin and xanthopterin. B. S. COOPER

See also Abstracts 59, 65, 189, 194.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

128. **The estimation of potassium in blood serum by the method of Kramer and Tisdall.** H. Chomse and I. Arend (*Hoppe-Seyl. Z.*, 1954, **297** [1], 45–46).—The value obtained depends on the concn. of K in the soln. to be estimated. The dilution brought about by the removal of proteins may lead to faulty results. If the volume used is reduced to 1 ml before precipitation, accurate results are obtained. A centrifuge tube marked at 1 ml is closed with a two-holed rubber stopper and dipped into a water-bath at 60° C while a stream of air is drawn through. The volume is reduced within a few min. and precipitation can then be satisfactorily carried out.
F. Powell

129. **A rapid method for the simultaneous determination of calcium and sodium in bone.** G. C. H. Bauer (*Acta Physiol. Scand.*, 1954, **31** [4], 351–358).—Samples (100 to 160 mg) of the fat-free dry powdered bone are shaken with 30 per cent. aq. trichloroacetic acid (3 ml) for 4 hr. Known amounts of $Na^{\cdot}$ and $Ca^{\cdot\cdot}$ are added to some of the samples before treatment with the acid, and the solutions are diluted to 100 ml before determination. Standard solutions are prepared from $Ca_3(PO_4)_2$ and NaCl in 30 per cent. aq. trichloroacetic acid; these contain approx. the same amounts of $Ca^{\cdot\cdot}$, $Na^{\cdot}$, $K^{\cdot}$, $Mg^{\cdot\cdot}$ and $PO_4^{\prime\prime\prime}$ as those expected in the samples. The Ca emission of the samples is then determined by means of a spectrophotometer with a flame photometer attachment; the emission of two standards, one containing more Ca and one less than the sample, is also determined. Standards are then selected that have the same Ca emission as the sample, but which contain various amounts of Na. The Na emission of the sample is then determined with two of the standards, one containing more Na and one less than the sample. From the values so obtained, the amounts of $Ca^{\cdot\cdot}$ and Na, in the sample are calculated.
J. N. Ashley

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monoximes. If a wide range of operation is desirable, the acetylbenzoyl procedure is best, but the diacetyl procedure is preferable in the range 0 to 50 mg per 100 ml. R. S. TONKS

136. **The determination of creatinine in plasma or serum, and in urine. A critical examination.** J. A. Owen, B. Iggo, F. J. Scandrett and C. P. Stewart (*Biochem. J.*, 1954, **58** [3], 426–437).—The Jaffé reaction for determination of creatinine in serum, plasma and urine is examined critically. The conditions under which the colour is developed must be carefully controlled. Temp. and time of colour development and measurement, and the pH of the medium are important factors. With the alkaline picrate reagent of Folin and Wu, the optical density of the colour obtained in a filtrate of pH $< 2{\cdot}5$, after 20 min. at 20° C and measured at 520 mμ, is directly proportional to amount of creatinine, up to a concn. of 400 μg per 100 ml. Both the "apparent" creatinine content of serum and the recoveries of creatinine added to serum are influenced by the method of pptn. of protein. Enzymic destruction of creatinine by NC-bacteria [a suspension of bacteria (sometimes called *Corynebacterium ureafaciens*)], or adsorption of creatinine on Lloyd's reagent (hydrated aluminium silicate), shows that the non-creatinine chromogen constitutes 20 and 5 per cent. of the total chromogen present in serum filtrates and diluted urine, respectively. Use of Lloyd's reagent with procedures that give "acid" filtrates of plasma or serum (pH $< 2{\cdot}5$), or with dil. urine, provides, at present, the most satisfactory method for determination of creatinine in these fluids. J. N. ASHLEY

137. **Elimination of *p*-nitrophenol in urine after action of the plant protective material E 605 [parathion].** S. von Eicken (*Angew. Chem.*, 1954, **66** [17–18], 551–553).—*p*-Nitrophenol appears in the urine as a fission product of E 605 (parathion) and can be detected by diluting the urine (1 + 10), acidifying it, and hydrolysing at 100° C and then extracting the nitrophenol with a 1 per cent. solution of *iso*pentanol in a mixture of light petroleum and ether (4 + 1), treating the extract with aq. 2 *N* NH_3, reducing with titanous chloride and converting the *p*-aminophenol formed to indophenol blue with *o*-cresol; the absorption of the dye is measured at 620 mμ. This test can also be applied to blood, but no positive results were obtained; apparently *p*-nitrophenol enters the blood only in cases of serious poisoning. R. C. MURRAY

138. **The estimation of barbituric acid derivatives in biological material for medico-legal purposes.** N. E. W. McCallum (*J. Pharm. Pharmacol.*, 1954, **6** [10], 733–739).—A rapid method is described for the estimation of barbiturates in blood and urine; it avoids the extraction of substances giving high values which are inherent in chloroform - sodium hydroxide procedures. A 90 per cent. recovery avoids calculations for losses, which are objectionable in forensic chemistry. Serum or plasma (2 ml) is placed in a separating funnel, 0·25 *N* NaOH (1 ml) is added and shaken with $CHCl_3$ (15 ml) for 3 min. The separated $CHCl_3$ layer is discarded, 0·5 *N* HCl (1·5 ml) and $CHCl_3$ (20 ml) are added and the mixture is shaken for 3 min. The $CHCl_3$ layer is run through a piece of cotton-wool (in a small funnel) into a second separator. Extraction is repeated in the first separator with further $CHCl_3$ (10 ml). The $CHCl_3$ in the second separator is then shaken with 0·1 *N* H_2SO_4 (7 ml) for 2 min., and the $CHCl_3$ layer is run into a third separator. Aq. 0·5 *N* NH_3 (10 ml) is added and shaken for 3 min. The aqueous phase is then centrifuged and the solution is compared in a spectrophotometer with the standard blank solution over the range 225 to 290 mμ. The standard blank is prepared by extracting the same volume of barbiturate-free serum. A modified procedure is described for urine. G. F. SOMERS

139. **Salicylamide. I. [Determination in blood serum].** J. M. Crampton and Elbert Voss (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43**, 470–473).—To 1 ml of serum is added 1 ml of 2 *N* HCl and any glucuronide is hydrolysed by refluxing gently for 3 hr. The mixture is extracted with 10 ml of ethylene dichloride for 5 min.; centrifuged for 3 min., and the supernatant layer is removed. To 9 ml of the decanted solvent layer are added 15 ml of 0·55 per cent. ferric nitrate soln. in 0·038 per cent. HNO_3 and the whole is vigorously shaken for 3 min. The optical density of the coloured layer obtained on centrifuging for 3 min. is read at 540 mμ, the salicylamide content being read from a standard curve. The method is applied to urine after dilution (up to 1 in 20), the natural phenol present causing little interference. G. R. WHALLEY

140. **Polarographic determination of thiol compounds in biological material. III. Polarographic behaviour of thiol compounds in serum and blood.** L. Jirousek and M. Petráčková (*Chem. Listy*, 1954, **48** [2], 260–264).—Direct polarographic determination of thiol compounds (**I**) in blood is possible only in deproteinised material. The total amount of **I** cannot be determined, but only a certain fraction of free **I**, which varies with the dilution of the blood with buffer soln. before deproteinisation. Measurements are thus limited to comparison studies under controlled standard conditions. A polarographic study of glutathione confirms the assumption that the polarographic inactivation of a part of **I** is due to combination. G. GLASER

141. **Rapid estimation of the serum vitamin B_{12} level by a microbiological method.** R. H. Girdwood (*Brit. Med. J.*, 1954, **ii**, 954–956).—Serum from 20 ml of blood collected aseptically was prepared for assay by the method of Rosenthal and Sarett (*J. Biol. Chem.*, 1952, **199**, 433) by using an acetate buffer of pH 4·6. The medium was that of Thompson *et al.* (*J. Biol. Chem.*, 1950, **184**, 175) modified by addition of thioglycollic acid and omission of ascorbic and fumaric acids. Certain batches of hydrolysed casein were found unusable owing to high blanks. Aseptic addition of the cyanocobalamin standard and diluted serum to the medium resulted in lower blanks than addition before autoclaving. Growth of the test organism *L. leichmanii* was estimated turbidimetrically after incubation at 37° C for 16 hr. Results showed that in general vitamin B_{12} deficiency is indicated by a serum level < 130 $\mu\mu$g per ml. H. F. W. KIRKPATRICK

142. **The determination of 17 : 21-dihydroxy-20-ketosteroids in urine and plasma.** R. H. Silber and C. C. Porter (*J. Biol. Chem.*, 1954, **210** [2], 923–932).—A rapid method is described for the determination of 17 : 21-dihydroxy-20-ketosteroids in urine and plasma. No special equipment is needed except micro cuvettes for the Beckman spectrophotometer. The method comprises four steps: (*a*) a 15-sec.

extraction of the urine or plasma with 5 vol. of $CHCl_3$, (*b*) a 10-sec. wash of the extract with dil. alkali, (*c*) a 15-sec. extraction of an aliquot of the $CHCl_3$ extract with an alcoholic phenylhydrazine - H_2SO_4 reagent and (*d*) heating the extract to 60° C for 30 min. and determination of the optical density spectrophotometrically. When used for determination of 17-hydroxycorticosteroids in plasma, 5 μg of hydrocortisone are added to each 10 ml of plasma. The method is also applicable to determination of glucuronide 17-hydroxycorticosteroids in urine after preliminary hydrolysis with β-glucuronidase. For practical purposes, values are expressed in terms of hydrocortisone, which is used as standard. The method will determine 6 μg per cent. of hydrocortisone with 95 per cent. confidence limits of ± 3 μg per cent. J. N. Ashley

143. **Conjugated steroids. V. Hydrolysis of total ketosteroids in urine.** I. B. Oneson and S. L. Cohen (*Proc. Soc. Exp. Biol. Med.*, 1954, **86** [4], 728–729).—Urine residues are prepared as for the hydrolysis of ketosteroid sulphates (*J. Biol. Chem.*, 1953, **204**, 245). Portions of the butanol residues equivalent to 100 to 200 ml of urine are hydrolysed by the addition of 10 ml of 1:4-dioxan containing 10 per cent. v/v of conc. HCl and heating in a steam-bath for 10 min. About 17 per cent. more steroids are formed by this method than by the standard HCl procedure, about 20 per cent. of the total steroids being of the beta type. Heating with an excess of acid or heating for a long period results in destruction of beta steroids.
H. F. W. Kirkpatrick

144. **A simplified method for the routine determination of pregnanediol and pregnanetriol in urine.** A. M. Bongiovanni and G. W. Clayton, jun. (*Bull. Johns Hopkins Hosp.*, 1954, **94** [4], 180–186).—Incubate a 50-ml aliquot of freshly collected 24-hr. urine with 15,000 units of glucuronidase and 10 ml of 0·5 *M* acetate buffer (pH 4·5) at 37° C for 24 hr. If high steroid levels are expected use proportionally less urine and reagents. Extract 3 times with one-third of the vol. of pure benzene and wash the benzene with 3 × 15-ml portions of *N* NaOH and 3 portions of water. Evaporate the benzene on a steam-bath in a gentle current of air. Prepare a column (11 mm diam.) with 12 g of alumina and benzene; dissolve the urine residue in 5 to 10 ml of benzene with warming and apply the solution to the column, washing the residue twice with 3 to 4 ml of benzene to ensure quant. transfer. Elute with: (*a*) 100 ml of 0·05 per cent. ethanol in benzene —the eluate is discarded; (*b*) 150 ml of 2·0 per cent. ethanol in benzene—the eluate contains the pregnanediol; and (*c*) 100 ml of 10·0 per cent. ethanol in benzene—the eluate contains the pregnanetriol. Evaporate 50-ml aliquots of (*b*) and (*c*) to dryness, add 5 ml of ethanol and again evaporate to remove last traces of benzene. Add 10·0 ml of conc. H_2SO_4, with swirling, to each residue and read the colour after 20 min. at 325, 390, 425, 440 and 460 mμ. Calculate the corrected reading at 425 mμ from the expression: O.D. at 425 − [(O.D. at 390 + O.D. at 460) ÷ 2]. This corrected reading obeys Beer's law over a wide range of concn. for both steroids although the true max. for pregnanetriol is at 440 mμ and that for pregnanediol is at 325 mμ. The relative purity of the residues may be assessed by taking these readings. The corrected $E^{1\%}_{1\,cm}$ at 425 mμ is 283 for pregnanetriol and 40 for pregnanediol. H. F. W. Kirkpatrick

145. **Determination of lactic acid in cultures of lactobacilli.** A. J. G. Barnett (*Nature*, 1954, **174**, 649).—Although the Barker and Summerson method for determining lactic acid in blood (*J. Biol. Chem*, 1941, **138**, 535) may be applied to determinations in both silage and milk, it cannot be used to determine the lactic acid present in cultures of lactobacilli. The Long method (*Biochem J.*, 1946, **40**, 27) gives satisfactory results and recovery tests indicate that material containing considerably larger quantities of lactic acid than those suggested by Long may be successfully handled. D. Liff

146. **The chromatographic determination of glucosamine with ninhydrin.** W. J. Payne and R. J. Kieber (*Arch. Biochem. Biophys.*, 1954, **52** [1], 1–4).—A modified partitioning solvent, *viz.*, pyridine - butanol - water (3:2:1·5), is proposed for the separation of glucosamine in the presence of uronic acids on chromatograms also carrying true sugars. With the proposed solvent, the constituents of polysaccharides can be determined without the necessity for a separate chromatographic procedure for the sugars. Glucosamine and other amine derivatives of sugars are detected by spraying the chromatograms with 0·05 per cent. ninhydrin in *n*-butanol and heating to 105° to 110° C for 10 min. in a hot-air oven. W. H. C. Shaw

147. **A comparison of the bronchodilator activities of adrenaline and noradrenaline. A proposed procedure for the biological assay of adrenaline solutions containing noradrenaline.** F. C. Lu and M. G. Allmark (*J. Pharm. Pharmacol.*, 1954, **6** [8], 513–521). — A precise and accurate method (described) based on the contraction and relaxation of the circular muscle of the tracheal preparation, which, when used in conjunction with the blood-pressure method, seems to be suitable for the assay of adrenaline solution containing 10 to 20 per cent. of noradrenaline. By this method, the mean bronchodilator activity of noradrenaline in terms of adrenaline was 5·0 to 10·3 per cent. J. Caley

148. **Determination of adenine.** R. Lecoq (*Ann. Pharm. Franç.*, 1954, **12**, 104–109).—The part played by adenine in the vitamin-B complex is discussed. Blood (from a fasting subject) is collected with oxalate or heparin and treated with trichloroacetic acid and filtered after 20 min. Neutralisation of the filtrate (NaOH and then acetic acid against phenolphthalein) followed by the addition of Na_2HPO_4 and then uranyl nitrate, mixing and standing for 20 min. affords a uranium - adenine complex ppt. that is separated by centrifuging and washed with aq. precipitant soln. Solution of the ppt. in aq. H_2SO_4 (by shaking, on a water-bath for 45 min.) followed by cooling and neutralisation with aq. NH_3 precipitates the uranium, which is separated by centrifugation. A portion of the supernatant liquor containing the adenine is mixed with buffer soln. and then treated with ammoniacal $AgNO_3$; the silver - adenine derivative is estimated by photoelectric or other turbidimetric methods. Details of quantities and solution compositions are stated.
F. R. Mumford

149. **The identification of spleen propionylcholine by infra-red microspectroscopy.** D. L. Wood (*Biochem. J.*, 1954, **58** [1], 30–31).—The i.r. spectra of acetylcholine and propionylcholine perchlorates are recorded. It is possible to distinguish between these two esters, and identify them in mixtures, by use of i.r. microspectroscopy. The acetyl ester

spectrum has a band at 8·05 μ that is absent from the spectrum of the propionyl ester, whilst this ester has strong absorption at 8·35 μ; with the acetyl ester, this is a region of max. transmission. The acetyl ester has a broad multiple band near 10·5 μ, and the propionyl ester a single sharp peak at 10·50 μ. J. N. ASHLEY

150. **Tetraphenyl diboroxide. III. A new method of detecting choline, acetylcholine and other quaternary ammonium compounds.** R. Neu (*Z. anal. Chem.*, 1954, **143** [1], 30–38).—Spot tests are described for detecting and distinguishing between choline, acetylcholine and other quaternary ammonium compounds, including the long-chain and other surface-active compounds. The unknown is mixed in turn with a mixture of tetraphenyl diboroxide and morin, rutin, quercetin, hyperin and quercitrin, and the colour obtained in ordinary and u.v. light is observed. The compound can then be identified by means of the extensive tables provided. Dodecyltriphenylphosphonium bromide and similar compounds also give colours. The reaction is sensitive; 1 μg of choline or 0·1 μg of acetylcholine can be detected. P. S. STROSS

151. **Estimation of adenosine triphosphate, adenosine diphosphate, adenosine monophosphate and inosic acid in resting and contracting frog rectus muscle.** G. Lange (*Experientia*, 1954, **10** [6], 268–269).—A method for the estimation of muscle nucleotides is described. The separation of nucleotides is accomplished by paper ionophoresis at high voltages as well as by paper chromatography. Even small changes in the nucleotide content, such as the changes in a single muscular contraction, can be well observed by this method. R. S. TONKS

152. **The determination of adenosine triphosphate by means of phosphoglyceric acid kinase.** B. W. Holloway (*Arch. Biochem. Biophys.*, 1954, **52** [1], 33–37).—The method is based on the catalysis by phosphoglyceric acid kinase of the reversible reaction between adenosine triphosphate (ATP) and 3-phosphoglyceric acid to give adenosine diphosphate (ADP) and 1:3-diphosphoglyceric acid. Quant. reaction is obtained by addition of hydroxylamine to convert 1:3-diphosphoglyceric acid as it is formed to 1-hydroxamic-3-phosphoglyceric acid, which is then determined as the coloured Fe^{III} complex. Under controlled conditions of incubation time and amount of enzyme, colour is proportional to concn. for 0·3 to 2·5 μ-moles of ATP. No reaction is given by pyrophosphate, creatine phosphate, phosphoenol pyruvate or ADP, but inosine triphosphate may show partial reaction. The preparation of the enzyme from pea-meal is described.
W. H. C. SHAW

153. **A method for enzymic determination of ATP [adenosine triphosphate] in tissues.** O. Forsander, B. Rehell and C.-E. Räihä (*Acta Physiol. Scand.*, 1954, **31** [2–3], 119–124).—A sample of tissue ($\simeq$ 100 mg) is homogenised with 2 per cent. aq. $HClO_4$. The mixture is then centrifuged, and the supernatant liquid is treated with a specific enzyme from potatoes (*cf.* Lee and Eiler, *Science*, 1951, **114**, 393), which hydrolyses the end phosphate group from the ATP; the liberated PO_4''' is determined photometrically at 740 mμ by the sodium molybdate - stannous chloride method, and the amount of P is ascertained from a standard graph. An aliquot of the residual protein left after centrifuging is analysed for N by the micro Kjeldahl method. The results are expressed as μg of ATP phosphorus per 100 mg of protein. J. N. ASHLEY

154. **An accurate method of amino-acid estimation suitable for component analyses on paper chromatograms.** H. Boser (*Hoppe-Seyl. Z.*, 1954, **296** [1–2], 10–18).—The amino-acids in 20 mg of protein hydrolysate can be separated and determined by a combination of paper electrophoresis and paper chromatography and the application of a micro modification of the copper phosphate method to the estimation of the acids so separated. Hydrolysis is carried out with 6 *N* HCl under reflux in a stream of CO_2. The residue is taken up in water to form an approx. 10 per cent. soln. A sample is taken with an R.B.C. pipette for total amino-N determination and with a haemoglobin pipette 60 μl is applied to paper for electrophoresis, which is carried out in Michaelis barbitone buffer at pH 5·5 for 3½ hr. at 265 V and 3 to 5 mA per cm width. Amino-acids are located with 0·02 per cent. ninhydrin and heating for 5 min. at 105° C. Sharply defined acids are cut out. The remainder is extracted with water and used for chromatography with phenol saturated with 0·30 per cent. aq. NH_3. Acids are located with 0·01 per cent. ninhydrin and 0·1 *M* phosphate buffer at pH 7·0 to 7·5, and are examined at 365 mμ. Of the original hydrolysate, 40 μl are also run as separate single-dimension chromatogram with butanol - acetic acid; location is as before. The individual acids are then estimated by the copper phosphate method and a table is given of corrections to be used for deviations in the case of individual acids from theoretical Cu to N ratio. F. POWELL

155. **Simple method of separating amino-acids by chromatography on paper.** N. M. Sisakyan, E. N. Bezinger, P. G. Garkavi and G. Ya. Kivman (*Dokl. Akad. Nauk, SSSR.*, 1954, **96** [2], 343–346).—Redfield's (*Biochim. Biophys. Acta*, 1953, **10**, 344) two-dimensional paper-chromatographic method has been simplified and the size of the paper required reduced, by using a mixture of alcohol, water and pyridine (40:10:2) in one direction, and a mixture of *n*-butanol, ethyl methyl ketone, water and diethylamine (20:20:10:2) in the other, and developing the spots with ninhydrin. Twenty amino-acids can be separated on a sheet measuring 28 cm × 28 cm, and occasionally on one 14 cm × 14 cm. R_F values are reported for the acids separated. R. C. MURRAY

156. **Quantitative paper-chromatographic determination of amino-acids.** A. M. Municio (*Rev. Cienc. Apl.*, 1954, **8**, 218–233).—Methods of visual comparison (approximate only), direct treatment to remove the ninhydrin-developed stains followed by colorimetric analysis, isotopic dilution, "development" of the stains and measurement of the areas covered after development, direct photometry of the stains and retention analysis (the developing solvent being chosen to react with the stain) are all compared and the techniques are described in detail, together with a few special methods of analysis. (119 references.) L. G. L UNSTEAD-JOSS

157. **The quantitative micro-analysis of free amino-acids by means of a simple copper-complex method.** H. W. Speir and G. Pascher (*Hoppe-Seyl. Z.*, 1954, **296** [3–4], 147–154).—Free amino-groups can be estimated by measuring the copper taken into solution in the form of a complex from an insoluble copper salt. One atom of copper is equivalent to two of N. This ratio only holds at pH 7·4. If a

higher pH is used, 1 atom of Cu combines with less than 2 atoms of N. Interference from other substances is also less at pH 7·4 and much lower blanks are obtained. A few amino-acids do not give the theoretical ratio even at this pH, and the reasons for this are discussed. *Procedure*—Add to the nearly neutral de-ionised soln. (1 ml), containing 50 to 550 μg of amino N, borate buffer pH 7·4 (9 ml) and a knife point of copper phosphate. Shake for 1 hr. and then centrifuge. To the centrifugate (1 ml), add 5 per cent. w/v of Na_3PO_4, 1 drop of blood serum and, after a time, 7 ml of distilled water containing 1 ml of 5 per cent. sodium diethyldithiocarbamate. Mix, and, after 5 to 10 min., measure the resulting yellow colour. F. POWELL

158. **The quantitative cleavage of benzyloxycarbonyl radicles from their amino-acid and protein derivatives and their micro-determination as toluene.** F. Micheel and B. Schleppinghoff (*Microchim. Acta*, 1954, [5], 494–500).—Cleavage of N-benzyloxycarbonyl radicles is effected by hydrogenation with Pd - $CaCO_3$ in water and the toluene formed is determined spectrophotometrically. The toluene is extracted from the aqueous phase with light petroleum (boiling range 30° to 45° C) in a special liquid - liquid extractor having mercury-sealed joints. The extract is diluted to give a toluene concn. between 1 in 1600 and 1 in 6000 and the extinction at 268·5 mμ is measured. Toluene adsorbed on the solid catalyst is extracted with 96 per cent. ethanol and determined separately. With N-benzyloxycarbonyl, 95 to 97 per cent. of the benzyloxycarbonyl radicles are obtained as toluene; with N-benzyloxycarbonylgelatin, the toluene found corresponds to 95 per cent. of the amount indicated by colorimetric determination of tyrosyl radicles. E. HAYES

159. **A manometric determination of nitrate reductase activity.** J. Robinson (*Arch. Biochem. Biophys.*, 1954, **52** [1], 148–150).—Nitrite formed by reduction of nitrate is determined manometrically in a conventional Warburg apparatus. The method is useful in the presence of pigments and high salt concentrations. *Procedure*—Place the test soln. (in 2 ml of 0·05 *M* phosphate buffer at pH 7·6) in the vessel and 0·2 ml of sulphamic acid soln. (4 g in 50 ml water mixed with 50 ml of cold 40 per cent. H_2SO_4) in the side-arm. After equilibration at 30° C, mix the two solutions and determine the N evolved. W. H. C. SHAW

160. **New methods in enzymology. III. Polarometric determination of cytochrome oxidase and succinic oxidase activities.** O. Pihar and L. Dupalová (*Chem. Listy*, 1954, **48** [2], 265–271).—A rapid polarometric determination of the activity of cytochrome oxidase (**I**) is based on the decrease of the peroxidic wave at 1·1 V with the dropping-mercury electrode. The activity of **I** was measured in the system oxidase - cytochrome-*c* (**II**) - *p*-phenylenediamine (**III**). In the presence of **III** only a small part of **II** is reduced. The concn. of ferrocytochrome (**IV**) was calculated from the concn. of **II** and **III** and normal potentials of both redox systems. The dependence of the activity of **I** on the actual concn. of **IV** is similar to that in the manometric determination with ascorbic acid. The polarographically obtained values of the activities of **I** and succinic oxidase in various organs of rats are compared with those obtained by other methods. G. GLASER

161. **Estimation and characteristics of the phosphohexose-isomerase of serum.** F. Bruns and K. Hinsberg (*Biochem. Z.*, 1954, **325** [7], 532–536).—The method described for the estimation of phosphohexose-isomerase in serum is based on the formation of fructose 6-phosphate from glucose 6-phosphate, and estimation of the former by a colour reaction with resorcinol (Roe, *J. Biol. Chem.*, 1934, **107**, 15). Serum (1·0 ml) is incubated for 30 or 60 min. with 0·1 *M* barbitone buffer at pH 7·8 (1·0 ml) and 0·06 *M* sodium glucose 6-phosphate at pH 7·8 (1·0 ml). Proteins are precipitated by the addition 7 per cent. trichloroacetic acid (7 ml). To 2 ml of the filtrate are added 0·1 per cent. ethanolic soln. of resorcinol (2 ml) and 30 per cent. HCl (6·0 ml). After heating for 8 min. in a water-bath at 80° C, the mixture is cooled for 5 min. in water and the extinction of the red soln. is determined at 490 mμ in a spectrophotometer. A blank determination is carried out, the substrate soln. being added after removal of the proteins. The activity is expressed as the amount of fructose 6-phosphate formed by 1 ml of serum under the standard conditions (1 micromole of fructose 6-phosphate = 260 μg = 22·4 cu. mm). G. W. CAMBRIDGE

162. **Spectrographic analysis of spores of *Bacillus megatherium*.** S. P. Cohen and D. A. Weiner (*Applied Spectroscopy*, 1954, **8** [1], 23).—Mn has been determined spectrographically in ashed samples of spores of *B. megatherium*. A preliminary qualitative examination provided results for the approximate metal content of the spores, and standards were made up with similar base composition; VCl_3 and $LiCO_3$ were used as internal standard - buffer, the lines Mn 2605·6 A and V 2687·4 A being used for the determination. Standards and samples were excited in under-cut cup graphite electrodes in a 12-amp. d.c. arc. The mean results for two samples analysed were 153 ± 22 p.p.m. of Mn and 112 ± 12 p.p.m. of Mn. B. S. COOPER

See also Abstracts 12, 34, 38, 174, 176, 224, 228.

Drugs

163. **A photometric determination of caffeine in medicinal preparations.** J. Richter (*Pharm. Zentralh.*, 1954, **93** [9], 339–340).—To 2 ml of dil. H_2SO_4 and 2 ml of 0·1 *N* iodine soln. in a sintered-glass crucible (porosity 4), 5 ml of the caffeine solution (0·15 to 0·9 mg per 100 ml) are added. After 1 min., suction is applied and the brown residue is washed free from iodine; after being sucked dry for 2 min., the residue is dissolved in 25 ml of methanol, the solution is filtered, the filter is washed with 25 ml of water and the light absorption of the combined filtrate and washing is measured in a suitable photometer with a 2-cm cell and an S47 filter; a blank determination is carried out. The amount of caffeine in mg per 100 ml = extinction × 44·4. Ethanol interferes and should be removed by heating; alkaloids, leptazol, local anaesthetics and pyrazoles also interfere, but acetanilide, acetylsalicylic acid, phenacetin, benzoic acid, salicylic acid, salicylamide and phenobarbitone do not. S. C. JOLLY

164. **Chemical estimation of digitalis.** J. M. Rowson (*Pharm. J.*, 1954, **173**, 71 and 88–89).—Chemical and physical methods for the assay of digitalis and its glycosides are reviewed. The best agreement with biological assays is given by the 3:5-dinitrobenzoic acid colour test. N. E.

165. **The assay of tincture of digitalis and of the glycosides of *Digitalis purpurea*.** H. Brindle, G. Rigby and Shri Nath Sharma (*J. Pharm. Pharmacol*, 1954, **6** [11], 887–893).—Chemical assay (with 3:5-dinitrobenzoic acid) did not agree with biological assay (B.P. 1953 method) for 8 commercial tinctures of digitalis. For decolorisation of tinctures before chemical assay, passage through an alumina column is recommended. Loss of chromogenic glycosides is < 10 per cent., and there is no development of a brown colour with the Kedde reagent such as may occur if the basic lead acetate treatment is used. No constituents other than those commonly known to be present in *D. purpurea* could be detected in the tinctures on examination by paper partition chromatography. A. R. ROGERS

166. **Comparative study of the assay of digitoxin by the U.S.P. XIII pigeon method and the U.S.P. XIV colorimetric assay.** H. A. Braun and L. M. Lusky (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [7], 440–442).—The U.S.P. XIV colorimetric method lacks the specificity necessary for the assay of digitoxin and its preparations, giving results in an appreciable number of instances up to 25 per cent. higher than the biological assay. J. CALEY

167. **Critical study of the official tests for digitoxoside [digitoxin].** R. Hazard, R. Charonnat and P. Lechat (*Ann. Pharm. Franç.*, 1954, **12** [2], 81–94).—The organoleptic characters, solubility, optical rotation, m.p., colour reactions and limits of H_2O stated in the various pharmacopœias are compared. For the water-limit test, the loss of water and the temp. and technique of desiccation are considered; it is found that the desiccated product rapidly re-absorbs H_2O and in 10 min. has regained most of the H_2O lost and is back to the original wt. in 1 hr. The residue on incineration and special tests are also considered. The variable results obtained with the U.S.P. XIV biological assay with pigeons are discussed. Determinations of toxicity to guinea pigs show similar discrepancies. The biological assays show no correlation with the physical variables, optical rotation and solubility in $CHCl_3$.
F. R. MUMFORD

168. **An *in vivo* method for the assay of heparin.** J. E. Jorpes, M. Blombäck and B. Blombäck (*J. Pharm. Pharmacol.*, 1954, **6** [10], 694–701).—A method is described for the determination *in vivo* of the anticoagulant activity of heparin in sheep. A polyethylene tube is inserted into the jugular vein and connected through a coupler and rubber tubing to a drip flask containing physiological saline solution. The saline solution is run into the vein at a rate of 15 to 20 drops per min. to prevent clot formation. The heparin solution is injected through the rubber tubing and washed in with saline. Only small doses (600 to 1100 units) of heparin are given and the maximum effect is reached in 4 min., when samples of blood are withdrawn from the vein after disconnecting the saline drip. The blood coagulation time is determined in a special chamber. A new dose is not given until the coagulation time has returned to normal (usually 4 hr.). Four or five animals are used concurrently and 10 per cent. differences in the doses of heparin can be detected. By this method the new Swedish national standard heparin contained 110 i.u. per mg compared with 108 i.u. per mg by the fresh whole-blood method, 107 i.u. per mg when assayed with thrombin and citrated plasma, 96·2 i.u. per mg by the U.S.P. XIV method and 103 i.u. per mg by the B.P. 1953 method. The former Swedish standard heparin contained 81 i.u. per mg by the fresh whole-blood method, 80·3 i.u. per mg by the described method, but only 67 i.u. per mg by the U.S.P. method. It is concluded that artificial systems involving citrated plasma and/or salted whole blood, including the U.S.P. and B.P. methods, are unreliable, the method with fresh whole blood *in vitro* gives results identical with those obtained *in vivo*.
G. F. SOMERS

169. **Determination of penicillin by the hydroxylamine method.** Poul Mørch (*Dansk. Tidsskr. Farm.*, 1954, **28** [8], 157–179).—The hydroxylamine method for the determination of penicillin (**I**) as modified by Boxer and Everett (*Brit. Abstr. C*, 1949, 404) has been investigated. The composition of the highly coloured ferric hydroxamate has been determined; the absorption max. is at 490 mμ and variation of $E^{1\%}_{1\,cm}$ with concn. of ferric salt reagent is reported. Fading of the colour is due entirely to reduction of the ferric salt with excess of hydroxylamine. The concn. of hydroxylamine should be ≮ 0·5 *N* if **I** hydroxamic acid formation is to be complete in a reasonable time. The addition of penicillinase (**II**) to the blank introduces two sources of error: (*i*) a vol. difference between test and blank and (*ii*) absorption at 490 mμ due to **II** itself; when strongly coloured **II** is used, the background absorption can be counteracted by adding **II** to the test solution immediately after adding the ferric salt. Many preparations of **I** cannot be assayed directly; purification and concn. can be effected by extracting the aqueous solution at pH 2 at 0° to 5° C with pentyl acetate, and then transferring **I** to 2 per cent. $NaHCO_3$ soln. By the specified experimental procedure, there is a constant loss of 3 per cent. S. C. JOLLY

170. **Bacteriological estimation of framycetin.** M. J. Phillipe, D. Benoist and M. F. Patte (*Ann. Pharm. Franç.*, 1954, **12** [5], 339–350).—For rapid (4-hr.) estimation of framycetin suitable for production control, a turbidimetric method is used in which 0·05 to 0·12 ml of a solution of framycetin sulphate (40 μg per ml) in 8 ml of a culture medium containing (1 per cent.), an 18-hr. culture of *Staphylococcus aureus* is used as a standard. The tubes are kept at 37° C for 3½ hr., the growth is then stopped with formaldehyde and the opacity is measured at 530 mμ on a photometer. A nearly linear plot is obtained of log. μg per tube against galvanometer reading. The results of the standardisation are statistically analysed, and a precision of 9·44 per cent. at the 0·95 probability level is attained. The more accurate but lengthy method of diffusion on an agar medium is also used. Preliminary tests show that the antibiotic diffuses more rapidly at higher pH and the estimates are carried out at pH 8, the highest compatible with a good growth of *Bacillus subtilis*. Aluminium boxes (10 × 30 cm) each with a plain Pyrex-glass plate on the bottom contain the medium. Fifty ml of medium are added and allowed to solidify, and 25 ml of the medium inoculated with *B. subtilis* are added and, when this is solid, paper discs (Vincent and Vincent, *Proc. Soc. Exp. Biol. Med.*, 1944, **55**, 162–164) dipped in the solution of framycetin and dried on filter-paper are placed on the medium. The culture is then incubated for 18 hr. at 37° C and 80 per cent. R.H. The diameter of the inhibited zones is then measured to 0·1 mm. A linear plot is obtained of log. concn. against diameter of the zone for the range 10 to 200 μg per ml. Variation of thickness of the agar

medium is overcome by the use of a Latin-square procedure for standard and test samples. Typical results and the method of calculation are given; precision for 9 determinations of each test sample or dilution of the standard is ± 5 per cent.
E. J. H. Birch

171. **Chemical methods for determination of methymycin in fermentation samples.** D. Perlman (*Antibiot. & Chemother.*, 1954, **4** [8], 859–863).—Investigations with diazotised sulphanilic acid, molybdo-arsenate, Folin - Ciocalteu and alcoholic alkaline dinitrobenzene (Zimmerman) reagents show that they all may be used for the determination of methymycin in plain aq. solutions with sensitivity decreasing in the order given. For fermentation samples, the method requiring Zimmerman's reagent is preferred in spite of its relatively low sensitivity, as its specificity is fairly high, particularly when used with the extraction procedure described. In addition, it gives results in good agreement with those obtained by a tube dilution method with *Klebsiella pneumoniae* and *Micrococcus pyogenes* as test organisms. Carbomycin, chlortetracycline, erythromycin and oxytetracycline possess functional groups similar to those occurring in methymycin and generally may also be determined in aq. solutions with the four reagents investigated. *Zimmerman procedure for methymycin*—Extract the fermentation sample (at pH 7·5) once with an equal vol. of pentyl acetate. Evaporate with a jet of air while heating on a steam-bath an aliquot of the solvent containing 160 to 3200 μg of methymycin to dryness in a test tube. Dissolve the residue in 0·5 ml of absolute ethanol. Add 0·5 ml of 2 per cent. *m*-dinitrobenzene in 95 per cent. ethanol and 0·5 ml of aq. 2·5 *N* KOH. Mix well and place in the dark for 30 min. Add 5 ml of 71 per cent. ethanol, mix and measure the colour, which is proportional to the concn. in the range given, against the reagents, using a 540-mμ filter. W. H. C. Shaw

172. **Observations on use of cylinders made from different materials in cylinder-plate method for testing antibiotics.** G. Nilsson and P. E. Nilsson (*Arch. Mikrobiol.*, 1954, **20**, 109–110).—The aluminium cylinders under test showed pronounced inhibitive effects on the growth of *Staphylococcus aureus*.
P. S. Arup

173. **Determination of phenolphthalein in chocolate preparations by non-aqueous titration.** S. Doernberg, M. Hubacher and I. Lysyj (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43**, 418–420).—The method proposed is based on a closed system that preserves a comparatively non-contaminating atmosphere and enables about 12 determinations to be made per day. A sample of chocolate containing 80 to 100 mg of phenolphthalein (**I**) is extracted with 3 × 5-ml portions of CCl_4 (to remove fat) and then 3 × 5-ml portions of acetone (to extract **I**). The acetone is removed on the water-bath, and 25 ml of (>98 per cent.) pyridine and 5 ml of anhydrous ethylenediamine are added and then 1 per cent. thymol blue indicator in pyridine. The solution is titrated slowly to a colour yellow to yellow-green (this is the blank) with a solution of 1·25 g of sodium in 100 ml of ethanolamine diluted to 500 ml with pyridine. The titration is then continued potentiometrically. The second titration figure × 0·0150 = wt. of phenolphthalein.
J. Caley

174. **The identification of barbiturates.** G. Hubner and E. Pfeil (*Hoppe-Seyl. Z.*, 1954, **296** [5–6], 225–228).—Protein-free liquids containing barbiturates are brought to pH 3 and extracted with ether. The ether is evaporated and the residue is dissolved in methanol and applied to paper for chromatography. Controls of 1 per cent. methanolic barbiturates are also applied. Descending chromatograms are developed with equal volumes of butanol and pentanol, which have been shaken with conc. aq. NH_3. The dried paper is sprayed with 1 per cent. ethanolic cobalt nitrate and dried at 80° C (1 to 2 min.) and then exposed to morpholine, piperidine or *iso*propylamine by laying the paper over a shallow dish containing the base and covering it with a glass plate. Violet spots of the cobalt complexes appear in a few minutes and are stable for a day or two; they can be restored by further exposure to the base. Limit of sensitivity is about 25 μg. Sulphonamides also react, but they are not extracted at pH 3. If it is necessary to detect sulphonamides, the ether extraction should be carried out at higher pH. They can also be detected with Ehrlich's aldehyde reagent. Unsaturated barbiturate derivatives give yellow spots on red-violet backgrounds when sprayed with 0·1 per cent. $KMnO_4$.
F. Powell

175. **Stability of barbiturates in solution. I. Determination of barbiturates by the cobaltammine reaction.** H. Nuppenau (*Dansk Tidsskr. Farm.*, 1954, **28** [9], 194–208).—The method of Holt and Mattson (*cf. Brit. Abstr. C*, 1950, 184) is applied with minor modifications to the determination of nine barbiturates. Calibration graphs with regression equations are given for colour measurements at 565 mμ; the graphs are linear for 1 to 5 mg of barbiturate per 25 ml of reaction mixture. Blank values are calculated by omission of the *iso*propylamine reagent. The reaction mixture must contain ≯ 0·1 per cent. of water. Results are not affected by the addition of ≃ 20 per cent. of possible hydrolytic products of the barbiturates. Further conditions for optimum accuracy are investigated, and detailed directions are given for the prep. of the anhydrous reagents and the standard samples.
P. S. Arup

176. **Determination of *iso*nicotinic acid hydrazide.** R. D. Strickland and W. Hentel (*Amer. J. Clin. Path.*, 1954, **24** [8], 988–992).—A gasometric method for determining *iso*nicotinic acid hydrazide levels in blood and urine is outlined. The method is based upon the fact that hydrazine and its derivatives release nitrogen quantitatively in the presence of saturated sodium iodate in alkaline solution. No interfering substances were discovered in serum or urine. For blood, the method involves the measurement of exceedingly small quantities of gas. In urine studies, the amounts of gas released are large enough to avoid difficulties in measurement. It is necessary that both the reagent and sample be degasified thoroughly before the determination.
R. S. Tonks

177. ***iso*Nicotinyl hydrazones as antitubercular agents, and derivatives for identification of aldehydes and ketones.** P. P. T. Sah and S. Anderson Peoples (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [9], 513–524).—*iso*Nicotinic acid hydrazide (isoniazid) (**I**) yields with a large number of carbonyl compounds crystalline hydrazones with distinct m.p. Three general methods of prep. are described. (*i*) To 0·7 g of **I** dissolved in 10 ml of water at 60° C, is added 0·006 to 0·01 g-mole of the carbonyl compound in 10 ml of pure ethanol, and the mixture is boiled gently for 5 min. and set aside at room temp. for

24 to 72 hr.; the cryst. hydrazones are removed by filtration, washed and recrystallised from methanol, or a mixture of methanol and benzene or light petroleum. (*ii*) For carbonyl compounds with b.p $< 100°$ C, 1·4 g of **I** are covered with 15 ml of the substance, and the mixture is shaken for 5 min. and then set aside for 16 hr.; the solvent is evaporated off and the residue is recrystallised. (*iii*) Hydrazones that are difficult or slow to crystallise should be converted to their hydrochlorides: to 0·0075 g-mole of the carbonyl compound dissolved in 15 ml of pure ethanol is added 0·7 g of **I** in 25 ml of pure ethanol, the mixture is boiled for 5 min. and, after standing and cooling, a mixture of conc. HCl (2 ml) and pure ethanol (8 ml) is added. After 24 hr. in a refrigerator, the crystalline hydrochloride is separated by filtration and recrystallised from boiling pure ethanol. The appearances and m.p. of 93 hydrazones and 14 hydrazone hydrochlorides are listed.

S. C. Jolly

178. **The reaction of aromatic aldehydes with pharmaceutical amino compounds. I. [Sulphonamides.]** C. G. Butler and P. H. B. Ingle (*J. Pharm. Pharmacol.*, 1954, **6** [11], 806–813).—The derivatives obtained by heating to boiling with a 5 per cent. soln. of salicylaldehyde in 95 per cent. ethanol, and allowing to cool, are recommended for the characterisation of sulphonamide drugs. A table gives the m.p., microscopic appearance, and time taken to ppt., for the salicylaldehyde derivatives of 13 sulphonamides. The benzaldehyde derivatives are less reliable. Sulphadimidine ($\simeq$ 20 mg) may be determined within $\pm$ 2 per cent. by heating for 15 min. with salicylaldehyde (1 g) in 95 per cent. ethanol (25 ml), and measuring the colour through an Ilford No. 601 filter. A. R. Rogers

179. **Complexometric determination of sulphate in pharmaceutical compounds.** J. S. Faber (*Pharm. Weekbl.*, 1954, **89** [41–42], 705–715).—A complexometric titration method is described for the determination of 40 to 80-mg amounts of SO_4''. To a quantity (w mg) containing 1 to 1·5 milli-equiv. of SO_4'' dissolved in 40 ml of water, 2 ml of NH_3 - NH_4Cl buffer solution (pH 10) are added, the mixture is heated to about 50° C and titrated with 0·1 N disodium ethylenediaminetetra-acetate (factor = t_c) to the blue colour of Eriochrome black T indicator (30 to 40 mg of a mixture of 1 part of dye to 100 parts of NaCl) (titre = a ml). To the titration liquid about 3 ml of 0·4 N HCl are added so that the colour just changes from blue to pink, the solution is heated to boiling, and 25 ml of 0·1 N $BaCl_2$ (factor = t_b) are added. After being heated over a low flame for 5 min. and set aside for 5 min., 5 ml of the buffer solution, 100 mg of magnesium dipotassium ethylenediaminetetra-acetate and, if necessary, 20 mg of the indicator are added, and the excess of $BaCl_2$ is titrated with 0·1 N disodium ethylenediaminetetra-acetate until the colour changes from red-violet to blue; 10 ml of 0·1 N $MgCl_2$ (factor = t_m) are added and the titration is continued immediately until the colour is again blue (titre = b ml). The percentage of metal ion = $(a \times t_c \times E \times 100)/w$, where E = equiv. wt. of the metal, and the percentage of $SO_4'' = 48 \times 100 \times (25t_b - b \times t_c + 10 \times t_m)/w$. The accuracy of the method is about 0·3 per cent. The complete assay of $MgSO_4$ and $ZnSO_4$ is possible by this method; it can also be applied to SO_4'' determinations on Na_2SO_4, K_2SO_4 and atropine sulphate. S. C. Jolly

See also Abstracts 12, 138, 147, 184, 199, 202.

Food

180. **Radio-isotopic dilution analysis for D-glucose and gentiobiose in "hydrol."** J. C. Sowden and A. S. Spriggs (*J. Amer. Chem. Soc.*, 1954, **76** [13], 3539–3541).—A radio-isotope dilution method is used to determine the content of D-glucose and gentiobiose (by use of ^{14}C-labelled sugars) in a representative sample of "hydrol" (a residual syrup obtained after the commercial crystallisation of D-glucose from acid hydrolysates of corn-starch). The value obtained for the D-glucose content agrees well with that obtained by a direct isolation method; the value for the gentiobiose content is much greater than that reported previously.

A. Jobling

181. **The determination of the total electrolyte concentration of sugar products.** J. Pomeranz and C. Lindner (*Anal. Chim. Acta*, 1954, **11** [3], 239–243).—Solutions (8 per cent. w/v) of brown sugar are passed through a column of Amberlite IR-120 cation-exchange resin and the eluates are titrated with 0·1 N NaOH, phenolphthalein being used as indicator. The titres are corrected for any original acidity of the soln. Thirty-two samples from various countries show an approx. linear relationship between the results obtained by this method and the results of ash determinations.

W. C. Johnson

182. **Simple chromatographic method for the determination of lactose in milk.** A. Nagabhushanam and K. V. Giri (*Curr. Sci.*, 1954, **23** [7], 221–223).—The procedure described involves (*i*) spotting an accurate amount (20 μl) of dil. milk (10 g diluted to 100 ml) on a Whatman No. 1 filter-paper and known amounts of standard lactose solution on either side of the milk spot, (*ii*) after spots have dried, developing the paper with a mixture of n-butanol, acetic acid and water (20:70:10), (*iii*) drying and treating with triphenyltetrazolium chloride (*cf.* Giri and Nigam, *Naturwissenschaften*, 1953, **40**, 343; *J. Indian Inst. Sci., A*, 1954, **36**, 49), and (*iv*) cutting the red bands of lactose, and extracting the colour with 95 per cent. ethanol and estimating it in a Klett - Summerson photo-electric colorimeter.

L. F. Taylor

183. **Loss of fat during souring of cream.** J. E. Roe, H. Edelson and W. E. Polzen (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 849–856).—Loss of fat arising from the souring of cream cannot be detected by the Babcock method over a 4-day period. The loss, as indicated by the Röse - Gottlieb method, is about 0·1 per cent. of the total percentage of fat per day over a 7-day period. A. A. Eldridge

184. **The determination of propylene glycol and glycerol in foods and medicinals.** A. P. Mathers and M. J. Pro (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 869–874).—Propylene glycol is distilled in a controlled-temperature still with Na formate and KOH; glycerol is distilled with mineral oil. A mixture is distilled with mineral oil, and the distillate is collected in two fractions. Sodium periodate is used for all oxidations, the amount used in the oxidation of propylene glycol being determined by differential titration with sodium arsenite after addition of $NaHCO_3$ and KI. Glycerol is determined after oxidation by titration with NaOH. Recoveries of propylene glycol and glycerol were, respectively, 98 to 101 and 97 to 101 per cent. A. A. Eldridge

185. **The determination of glycerol and propylene glycol in desiccated coconut by distillation with Decalin.** J. C. M. Griffin (*J. Ass. Off. Agric.*

Chem., 1954, **37** [3], 874–880).—The glycerol and propylene glycol are extracted with lime water, a 25-ml aliquot containing $\gtrless$ 50 mg of propylene glycol or 35 mg of glycerol is distilled with 50 ml of Decalin (decahydronaphthalene) in a Dean and Stark apparatus for a period of 2 hr. after all water has distilled over. The aq. layer is diluted to 100 ml. To one 40-ml aliquot are added 35 ml of 0·02 *M* KIO_4; after dilution to 100 ml and standing for 1 hr. $NaHCO_3$ (1 g), KI (0·5 g) and starch soln. are added. The liberated iodine is titrated with 0·02 *N* $KAsO_2$ (standardised against the KIO_4 soln.). Another 40-ml aliquot is neutralised with 0·02 *N* NaOH to bromo-cresol purple, and 35 ml of 0·02 *M* KIO_4 are added; after dilution to 100 ml and standing for 1 hr., 0·5 ml of propylene glycol is added; after a further ½ hr. the excess of KIO_4 has been destroyed and the acidity is determined with 0·02 *N* NaOH. A correction is made for the acidity of the KIO_4 soln. Then mg of glycerol = [(total ml of 0·02 *N* NaOH − (ml of 0·02 *N* NaOH required for KIO_4 acidity)] × 1·84; mg of propylene glycol = (ml of 0·02 *N* $KAsO_2$) − [4 (total ml of 0·02 *N* NaOH)] × 0·76. After corrections have been applied for the coconut blank and the acidity of the KIO_4, the recoveries of propylene glycol were from 95 to 99·9 per cent. and of glycerol about 105 per cent. N. E.

186. **Determination of furfural, pentoses and pentosans in distilled spirits.** A. P. Mathers and J. E. Beck (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 861–869).—A flask (with reflux condenser) holding the solvent for maintaining a constant temperature contains the reaction chamber fitted with a condenser and a steam inlet tube attached to a steam generator. Boiling xylene ensures that the temperature does not rise above the max. permissible. Furfural is determined spectrophotometrically, although other substances may also absorb light at 277 mμ. For the decomposition of pentosans and pentoses, HCl or a mixture of HCl and diethylamine is preferred. It is tentatively assumed that 81 per cent. of pentose is converted into furfural. A. A. ELDRIDGE

187. **Ester determinations in distilled liquors with increasing alkali concentrations.** R. L. Schoeneman (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 921–922).—An excess of alkali for hydrolysis ("Methods of Analysis," A.O.A.C., 1950, 9·17) of 5 to 10 ml of 0·1 *N* NaOH results in variations of ester content no greater than 2·6 g per 100 litres. Less than 5 ml of excess results in variations up to 11·0 g per 100 litres. A. A. ELDRIDGE

188. **Determination of traces of iron in wine.** L. Deibner and H. Bouzigues (*Mikrochim. Acta*, 1954, [5], 501–508).—The sample is treated with 110-volume H_2O_2 and then evaporated to small volume on a sand-bath. A slight excess of aq. NH_3 is added to the cooled residue, and the hydroxide ppt. formed is dissolved in a known volume of *N* HCl. An aliquot of this solution is treated with quinol and Na_2SO_3 in the presence of *o*-phenanthroline, and ammonium acetate is added to bring the pH within 4·8 and 6. The optical density of the soln. is then read in a photo-electric colorimeter fitted with a Wratten No. 75 blue-green (460 to 520-mμ) filter. The limits of error of the method are ± 5 to 10 per cent., depending on the Fe content of the sample. The method is rapid and phosphate does not interfere. E. HAYES

189. **The paper-chromatographic identification of eugenol and anisaldehyde in dessert wine and wine-containing drinks.** F. H. Mühlberger (*Z. anal. Chem.*, 1954, **143** [1], 21–30).—Eugenol and anisaldehyde are separated from vanillin and other aromatic components of wine by ascending paper chromatography with water-saturated light petroleum as solvent. The exact conditions (critical) are fully described. Although the eugenol and anisaldehyde have almost the same R_F value, they are distinguished by their reactions. Anisaldehyde gives with hydrazine a light-green strongly fluorescent hydrazone and a yellow colour with benzidine, whilst eugenol gives a blue-black colour with ammoniacal silver nitrate. After suitably concentrating the solutions, 1 to 2 mg eugenol and anisaldehyde per litre can easily be detected, although it is possible to detect as little as 0·2 mg per litre. Examination of a number of wines showed both compounds to be absent. Their presence therefore probably indicates adulteration. P. S. STROSS

190. **The chemical determination of bromoacetic acid in must, fruit juices, beer or wine.** A. Hansen (*Z. anal. Chem.*, 1954, **143** [1], 17–21).—The sample (50 to 100 ml) is extracted with ether for 24 hr. in a continuous-extraction apparatus with a receiver containing 2 *N* KOH soln., which converts the extracted bromoacetic acid to KBr. To the KOH - KBr - ether mixture, add MgO (10 mg), transfer to a porcelain dish, evaporate to dryness and carefully ignite at 475° to 500° C. Dissolve the residue in water, adjust to pH 5 with H_2SO_4, and after 15 min. make up to 25 ml. Test for presence of bromide with chloramine and fluorescein. If bromide is present, oxidise it to bromate with sodium hypochlorite in a pH 6·5 buffer and determine it iodimetrically. One mg of bromoacetic acid per litre can be determined with an accuracy of about 5 per cent. P. S. STROSS

191. **A separation of the triphenylmethane food colours by column chromatography.** G. G. McKeown and J. L. Thomson (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 917–920).—An aqueous solution of the dye, acidified with acetic acid, was passed through an alumina column and excess of acid was washed out; the colours were developed with aqueous pyridine, the excess being removed by washing first with water and then with dilute acetic acid. The positions of the colours were: Fast green FCF (FD & C green No. 3) 20 to 28 mm, Light green SF yellowish (FD & C green No. 2) 22 to 34 mm, Benzyl violet 4 B (FD & C violet No. 1) 36 to 50 mm, Guinea green B (FD & C green No. 1) 55 to 75 mm, Brilliant blue FCF (FD & C blue No. 1) 56 to 72 mm. Good separations of Brilliant blue FCF and Light green SF yellowish were attained. A. A. ELDRIDGE

192. **Studies on coal-tar colours. XVI. FD & C red No. 4.** C. Graichen and K. S. Heine, jun. (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 905–912).—The preparation of FD & C red No. 4 [2-(5-sulpho-2:4-xylylazo)-1-naphthol-4-sulphonic acid, Ponceau SX] and of its 6-sulpho isomer are described. Absorption curves for both substances and for 2:4-xylidine-5- and 2:4-xylidine-6-sulphonic acids are given. Both the spectrophotometric and the $TiCl_3$ titration procedures afford satisfactory analytical results. A. A. ELDRIDGE

193. **Studies on coal-tar colours. XVII. Ext. D & C red No. 11.** R. N. Sclar and K. A. Freeman (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 913–917).—Absorption curves for Ext. D & C red No. 11, prepared by coupling diazotised aniline with 8-acetylamino-1-naphthol-3:6-disulphonic acid, are

given; Beer's law is applicable to solutions containing 5 to 12 mg per litre. Chromatrope 2R, acid Fuchsin D and Ext. D & C red No. 11 can be separated by paper chromatography. All the samples examined contained Chromotrope 2R (< 0·5 per cent.) and acid Fuchsin D (0·9 to 2·0 per cent.). A. A. ELDRIDGE

194. **The determination of copper in oils and fats by means of dibenzyldithiocarbamic acid and its salts.** D. C. Abbott and R. D. A. Polhill (*Analyst* 1954, **79**, 547–550).—A method is described for the rapid absorptiometric determination of Cu (0·02 to 2 p.p.m.) in oils and fats. The sample (≃ 20 g) is first heated in a silica flask until its vol. is reduced to 1 or 2 ml; destruction of organic matter is then continued by a wet ash process. The diluted residual liquid is heated with Na_2SO_3 to remove NO_2 and is then extracted with a soln. of a colour reagent in CCl_4. The colour reagent may be dibenzyldithiocarbamic acid or its Zn, K or dibenzylammonium salt. The preparation of these salts is described. The absorption of the extract is measured at 435 mμ and the Cu content is ascertained from a calibration graph prepared by the same procedure. Interference by other commonly occurring metals is negligible and recovery is good. A. O. JONES

195. **Quantitative infra-red analysis of mixtures of [fatty acids].** I. R. C. McDonald (*Nature*, 1954, **174**, 703–704).—Differential analysis of mixtures can be used in infra-red solution analysis when a double-beam instrument is available. The effect of interfering substances can be eliminated by adding the interfering substance to the compensating cell, or a substance having an absorption peak at the same frequency as the interfering substance can be used. Solvent absorption is eliminated by having low concentrations and using either matched cells or a variable-space cell of thickness similar to that of a fixed-thickness cell. The method has been used to determine the percentage of trans unsaturated fatty acids in the presence of cis unsaturated fatty acids and saturated fatty acids. The method may also be used as a criterion of purity. A. J. MEE

196. **Determination of resinous substances in the presence of fatty acids.** K. Metzl and J. Mrva (*Chem. Listy*, 1953, **47** [7], 1099–1100).—The resin acid content of fats is determined by direct isolation and weighing in 2 ways. (*i*) Saponify the sample (1 g) by heating its soln. in toluene (30 ml) with a suspension of Ag_2O (2 g) away from direct sunlight. Strip the solvent, dry the residue for 1 hr. at 50° to 60° C, extract with dry ether, filter, evaporate the filtrate and decompose the silver salts of the resin acids by boiling with 6 *N* HCl for 30 min. Isolate the free acids by extracting with ether and evaporating to dryness. Weigh after drying at 100° to 105° C. (*ii*) Saponify the sample with 10 per cent. NaOH diluted with 6 vol. of water, evaporate, dry the residue at 100° to 105° C and weigh it. To an aliquot (2 to 4 g), add absolute ethanol and heat at 80° C on a steam-bath in a closed flask. After cooling, add dry ether (100 ml) and set aside for 24 hr. In this way only the sodium salts of the resin acids remain in soln. Decant, wash the residue with a (5 + 1) mixture of ether and ethanol, remove the solvents from the combined extracts, decompose the residue with HCl and isolate the free acids as in first method. G. GLASER

197. **Standardisation of alumina adsorbents for vitamin-A chromatography.** J. B. Wilkie and S. W. Jones (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 880–887).—Procedure for determining the adsorptive activity of Al_2O_3, by use of the dye FD & C yellow No. 4, is described. For different samples of Al_2O_3, the dye retained varied between 0 and 92·5 per cent. Samples prepared by heating at 620° C and subsequently treated with water had a retention of 28 and 34 per cent. and gave optimum performance in the chromatographic column. The used material can be rejuvenated by treatment with HCl, followed by washing and drying. Results for dye retention and vitamin-A content of margarine are tabulated for various combinations of Al_2O_3 and alkaline Al_2O_3. A. A. ELDRIDGE

198. **A chromatographic - spectrophotometric method for determination of vitamin A in margarine.** L. Rosner and H. Kan (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 887–894).—Margarine is saponified and extracted with ether, the residue from the evaporation of the ether being dissolved in light petroleum and passed through a column of alkali-treated alumina. Vitamin A is eluted with ether dissolved in light petroleum, successive portions containing increasing amounts of ether. Solvent is removed from the vitamin-A fraction with N, the residue is dissolved in *iso*propanol, and the vitamin A is determined spectrophotometrically at 325 mμ. A. A. ELDRIDGE

199. **The spectrophotometric determination of vitamin D in pharmaceutical preparations. I. Solution of calciferol B.P.** A. R. Rogers (*J. Pharm. Pharmacol*, 1954, **6** [11], 780–783).—The reagent of de Witt and Sullivan (*Brit. Abstr. C*, 1946, 186), concontaining $SbCl_3$ (20 per cent.) and acetyl chloride (2 per cent.) in ethylene dichloride, is added to an ethylene dichloride soln. of the unsaponifiable matter extracted from the sample, and the extinction at 500 mμ is measured. The effect of sterols in the oil base is eliminated by taking readings at 30-sec. intervals for 6 min. and extrapolating to zero time. The method determines calciferol in olive or arachis oil over the range 2000 to 4500 units per g within ± 3 per cent. A. R. ROGERS

200. **The plate assays of vitamins of the B group.** A. Jones (*Analyst*, 1954, **79**, 586).—In previous work (*Brit. Abstr. C*, 1949, 409; 1951, 55; 1952, 113; 1953, 318) on plate assays for thiamine, vitamin B_6, inositol, nicotinic acid, pantothenic acid and biotin in yeasts and yeast products, the use of five plates for the standard and five for each test sample with four or five levels of standard or sample in each plate was recommended. Recently the more normal (2 + 2) assay design has been successfully used. With test material of approx. known vitamin content, widely spaced standards can be used, the dose levels being arranged to lie well within the linear portion of the dose - response curve. Riboflavin in tablets and yeast extracts has been assayed by a plate method with the basal medium previously reported (*Brit. Abstr. C*, 1950, 608) for a 17-hr. tube assay. One ml of a suspension of *Lactobacillus helveticus*, with opacity equivalent to that of tube No. 5 of the Wellcome series, was used as inoculum for each 20 ml of basal medium. The levels of reference standard used in these assays are given with indications of the precision. A. O. JONES

201. **Separation and determination of vitamin-B_6 group. Separation by paper electrophoresis of riboflavine and of nicotinamide groups.** N. Siliprandi, D. Siliprandi and H. Lis (*Biochim. Biophys. Acta*, 1954, **14** [2], 212–218).—Previous separation of a mixture of all forms of vitamin B_6 has not been achieved either biologically or chemically. A

mixture containing about 10 μg of each of the vitamin-B_6 groups can be separated by paper electrophoresis in acetate buffer, pH 5·1, $\mu = 0{\cdot}05$, with a current of 3·5 mA for 6 to 7 hr. The spots are developed with diazotised *p*-aminoacetophenone. Pyridoxal phosphate and pyridoxamine phosphate move towards the anode, the former having the greater mobility; pyridoxamine, pyridoxin and pyridoxal migrate towards the cathode in decreasing order of mobility. No breakdown of the phosphate esters occurred. For quant. estimation the spots may be located by u.v. light and the materials can be eluted with 0·05 *M* acetate buffer and the extinction coeff. determined at 325 mμ. Larger amounts of these compounds may be separated by starch-column electrophoresis and the separation of pyridoxin, pyridoxal and pyridoxamine achieved by starch-column chromatography. Riboflavine (0·6 μg), flavine mononucleotide (1 mg) and flavine adenine nucleotide (5 μg) can be separated by the same paper-electrophoretic technique and the spots can be localised by their bright yellow fluorescence under u.v. light. Similarly, nicotinamide, diphosphopyridine nucleotide (DPN) and triphosphopyridine nucleotide (TPN) (15 μg of each) can be separated, and, after spraying with a (1 + 1) mixture of 2 per cent. $Na_2S_2O_3$ soln. and 4 per cent. $NaHCO_3$ soln. to convert DPN and TPN to their dihydro-forms, the spots are revealed as feebly fluorescent under u.v. light (impurities, one of which is probably adenosine diphosphate, are revealed as dark spots). By subjecting a mixture of vitamin B_2, vitamin B_6 and nicotinamide groups to paper electrophoresis for 10 hr. and localisation of the spots first with u.v. light, then with u.v. light after spraying with dithionite - bicarbonate mixture and finally with u.v. light after spraying with diazotised *p*-aminoacetophenone, all compounds can be identified.

G. W. CAMBRIDGE

202. **The microbiological determination of vitamin B_{12} utilising a mutant strain of *Escherichia coli*.** F. J. Bandelin and J. V. Tuschhoff (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [8], 474–477).—By using a simple turbidimetric method for the assay of vitamin B_{12} with a new *E. coli* 113-3 mutant, the results obtained with cyanocobalamin standards and various tissues and extracts compared favourably with those obtained by the *L. leichmanii* method. Growth response is affected by shaking during incubation period; tubes not shaken show a steeper growth response curve, reaching a max. in a shorter time period. Methionine interferes if present in concn. of more than 70,000 times that of the vitamin. G. R. WHALLEY

203. **Use of *p*-chloromercuribenzoic acid in the determination of ascorbic acid in the presence of sulphydryl compounds [thiols].** J. A. Owen, B. Iggo and D. B. Horn (*Nature*, 1954, **174**, 701).—H_2S, cysteine and related thiols may, in certain circumstances, react with indophenol and may interfere with the determination of ascorbic acid with this reagent. *p*-Chloromercuribenzoic acid reacts with thiols, but does not react with indophenol, and addition of this substance completely suppresses the decolorisation of indophenol by H_2S, cysteine and glutathione. *p*-Chloromercuribenzoic acid may be of value in increasing the specificity of the indophenol reaction applied to the determination of ascorbic acid in biological material containing thiols. It has advantages over mercuric acetate for the removal of cysteine and other compounds because addition of H_2S to remove the excess of Hg^{++} and to convert the dehydroascorbic acid formed into ascorbic acid is unnecessary. A. J. MEE

See also Abstracts 12, 17, 141, 233.

Sanitation

204. **Spectrophotometric determination of traces of copper in water.** L. Ghyssaert (*Bull. Cent. Belge Étud. Docum. Eaux*, 1954, [23], 56–60).—The method of determining Cu in water by forming the complex with 2:2′-diquinolyl, extracting this with *iso*pentanol and determining the absorption of the solution is described briefly. Studies of various solvents in place of *iso*pentanol are described. Trichloroethanol was the most suitable; it is heavier than water, thereby facilitating extraction, and is readily prepared pure. The partitition coeff. of the complex is 1600 in *iso*pentanol and 384 in trichloroethanol. Max. absorption occurs at 550 mμ. Experimental details are given. The mean error is 0·5 per cent.

S. C. I. ABSTR.

205. **Determination of iron in [natural] waters.** R. Buydens and R. Muylle (*Bull. Cent. Belge Étud. Docum. Eaux*, 1954, [23], 52–55).—In this further description (*cf. Bull. Cent. Belge Étud. Docum. Eaux*, 1952, [18], 241) of spectrophotometric methods of determining iron in natural waters, results with thioglycollic acid or disodium 1:2-dihydroxybenzene-3:5-disulphonate are compared with those with thiocyanate, 2:2′-dipyridyl or *o*-phenanthroline, and are found to be less sensitive. Of the last three of these, *o*-phenanthroline gave low results and by its use iron was incompletely recovered. With thiocyanate the pH must be less than 0·5, there must be no excess of H_2O_2 and there must be present 3 ml of $(NH_4)_2S_2O_8$ or HNO_3 per 100 ml of water. For determining iron in complexes with organic compounds, the sample is evaporated to dryness in a platinum crucible and heated to redness for 3 min. The residue is dissolved in HNO_3 and diluted, and the Fe is found as before. Results are higher than by direct oxidation. S. C. I. ABSTR.

206. **Spectrographic techniques as applied to the analysis of sea water.** R. D. Hitchcock and W. L. Starr (*Applied Spectroscopy*, 1954, **8** [1], 5–17).—For the estimation of cations in sea water without preliminary chemical separation, various spectrographic methods are applicable. Direct excitation of the solution may be carried out by the rotating-disc method, the porous-cup method or by flame excitation. For elements present in low concentration, residue excitation methods are necessary, such as, the impregnated-electrode method, filled-crater method or briquette method. The rotating-disc method, supplemented by flame photometry, gave satisfactory results for Na (10,000 p.p.m.), Mg (1170 p.p.m.), Ca (396 p.p.m.), K (373 p.p.m.) and Sr (8·3 p.p.m.). Briquette excitation was suitable for Al (0·58 p.p.m.). All these results are in good agreement with other values reported in the literature. It was also found that Ni (0·23 p.p.m.) could be estimated by the briquette-excitation technique in sea water samples studied (from the harbour at Port Hueneme, California); literature values for nickel concentration are weaker by several orders of magnitude than the above value. B. S. COOPER

207. **The rapid determination of calcium and magnesium in sea water.** A. de Sousa (*Anal. Chim. Acta*, 1954, **11** [3], 221–224).—For purposes of marine biology there is need of a rapid method of Ca^{++} and Mg^{++} determination suitable for use at sea. The following method can be completed in 1 hr. and

the slight traces of $Fe^{\cdots}$ and $Al^{\cdots}$ usually present in sea water are without significant effect. *Procedure*—To 100 ml of sea-water, add aq. NaOH until the pH is slightly greater than 12, add saturated aq. murexide to produce a strong red colour and titrate with 0·1 *M* complexone III to a blue-violet colour (1 ml of 0·1 *M* complexone III ≡ 4·008 mg of Ca). To another 100-ml portion, add 1 ml of a 0·4 per cent. w/v soln. of Eriochrome black T in ethanol and 10 ml of an aq. buffer soln. containing 54 g of NH_4Cl and 350 ml of aq. NH_3 (25 per cent.) in 1 litre; heat the soln. to 40° to 50° C and titrate with 0·1 *M* complexone III until the indicator changes from red to blue. The second titration gives the concn. of $Ca^{\cdot\cdot}$ plus $Mg^{\cdot\cdot}$. Calculate the $Mg^{\cdot\cdot}$ by difference. One ml of 0·1 *M* complexone III ≡ 2·432 mg of $Mg^{\cdot\cdot}$. W. C. JOHNSON

208. **Determination of synthetic detergents in sewage.** G. P. Edwards and M. E. Ginn (*Sewage Ind. Wastes*, 1954, **26** [8], 945–953).—At least 90 per cent. of synthetic detergents used in the United States are of the anion-active type and are alkylarylsulphonates. The Lewis and Herndon modification (*Brit. Abstr. C*, 1953, 442) of Barr's method has proved satisfactory for determination of these detergents. It was found that better separation of the emulsion took place with a solvent lighter than water, so commercial hexane was selected. The pH is kept constant between 7·0 and 7·5 by the use of a phosphate - disodium dihydrogen ethylenediaminetetra-acetate buffer, which avoids errors caused by the presence of calcium and magnesium salts. Bromophenol blue or Azo phloxine can be used as the indicating dyestuff. Full instructions for the method are given. A. WEBSTER

209. **Apparatus for the determination of high concentrations of methyl bromide [used] in fumigation.** D. Loveday (*J. Sci. Food Agric.*, 1954, **5** [8], 376–381).—An apparatus for determining concentrations of methyl bromide in air over the range 0 to 35 mg per litre is described and illustrated. It is based on the method previously described by Call (*Brit. Abstr. C*, 1953, 277), which depends on the length of coloured stain produced by interaction of Br, as a combustion product of methyl bromide, with paper impregnated with fluorescein. In the apparatus described, however, a swept-sample technique is used to obtain a representative sample, and buffered phenol red is used as indicator. O. M. WHITTON

210. **Ultra-violet and infra-red spectrophotometric analysis of an insect repellent mixture.** W. A. Sassaman and S. P. Cohen (*Applied Spectroscopy*, 1954, **8** [2], 67–70).—By means of absorption measurements in the ultra-violet at 227·5 mμ and 280·3 mμ, and in the infra-red at 8·99 μ and 9·60 μ, the concentrations of N-butylacetanilide, benzyl benzoate, 2-butyl-2-ethylpropane-1:3-diol and Tween 80 (polyoxyethylenesorbitan mono-oleate) can be determined in a mixture of the four components. B. S. COOPER

211. **Problems encountered in physicochemical determination of Warfarin [3-(α-acetonylbenzyl)-4-hydroxycoumarin].** F. B. Coon, E. F. Richter, L. W. Hein and C.H. Krieger (*J. Agric. Food Chem.*, 1954, **2** [14], 739–741).—The rodenticide is analysed spectrophotometrically by determining the absorption of its alkaline solution at 308 mμ. Some baits prepared by a coating process give poor recoveries when the initial extraction is carried out with ether; exact duplicate blanks are also needed, and some baits contain interfering materials. More satisfactory results are obtained with less interference by extracting from the bait with dil. NaOH or sodium pyrophosphate. After acidification, the compound is extracted into ether and estimated as in the original method. Results obtained by the 2 methods and by biological assay in rats are compared. Chromatography on silicic acid or Attapulgus clay eliminates interfering substances, but some clays cause loss of the rodenticide. C. E. SEARLE

See also Abstract 113.

Agriculture and Plant Biochemistry

212. **Determination of nitrogen in agricultural materials by the Nessler reagent. II. Micro-determinations in plant tissue and in soil extracts.** S. H. Yuen and A. G. Pollard (*J. Sci. Food Agric.*, 1954, **5** [8], 364–369).—Direct Nesslerisation procedures are given for micro-determination of N in H_2SO_4 - H_2O_2 digests of plant tissue, with accuracy up to 5 per cent., and of ammonia-nitrogen in soil extracts and soil solutions, with somewhat greater errors. In a spectrophotometric study of the Nessler colour, a filter showing max. transmission at 420 mμ absorbs most of the colour. In the H_2SO_4 - H_2O_2 digestion of plant tissue, added NH_3 is fully recovered, but 23 to 50 per cent. of added nitrate is lost, the relative loss increasing with the amount of nitrate added. Bromothymol blue is a suitable internal indicator for neutralising test solutions before Nesslerisation. The decolorisation of soil solutions and soil extracts by activated charcoal treated with NaCl is described. This charcoal does not absorb much NH_3 from soil extracts made with Morgan's reagent, 2·5 per cent. acetic acid or H_2O. For more accurate determination of NH_3 in soil extracts, distillation of the NH_3 into acid in the presence of bromothymol blue followed by Nesslerisation is preferable. O. M. WHITTON

213. **One-step digestion procedure for the estimation of total phosphorus and nitrogen in mould tissue.** S. P. Damle and P. S. Krishnan (*Anal. Chim. Acta*, 1954, **11** [3], 225–228).—For the determination of total P in mould tissue the material is digested with H_2SO_4 and H_2O_2. It is now shown that the same digestion, modified as follows, can be used for the determination of total N. Mix 0·25 to 0·5 g of dried or acetonised mould tissue with 4 to 6 ml of H_2SO_4 in a 50-ml Kjeldahl flask and heat gently until the material dissolves. Heat strongly until fumes appear, cool for 15 to 30 sec. and add 2 to 4 drops of Superoxol (30 per cent. H_2O_2 soln.) down the side of the flask. Continue heating and add 2 to 4 drops of Superoxol every 15 min. until the soln. is colourless and continue heating for 30 min. (total time ≃ 6 hr.). Cool, dilute with water and boil for 3 min. The method is applied to the determination of N in 9 different samples of 5 different moulds, also to acetonised liver, ground-nut cake and acetonised *B. cadaveris*; it gives results in fair to good agreement with those obtained when Se, $CuSO_4$ and K_2SO_4 are used to catalyse the oxidation. The second method requires a longer digestion time (8 to 10 hr.). W. C. JOHNSON

214. **Xanthophyll determination in dehydrated alfalfa [lucerne] meal.** E. M. Bickoff, A. L. Livingston, G. F. Bailey and C. R. Thompson (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 894–902).—The rehydration procedure for the determination of carotene (Bickoff *et al.*, *Brit. Abstr. C*, 1953, 219) is applicable

to xanthophyll, which is eluted with a mixture of hexane and ethanol. The absorbance of this solution, and of the hexane - acetone eluate of carotene, may be read in a colorimeter with a 440-mμ filter, or the carotene and xanthophyll may be determined spectrophotometrically at 451 and 445 mμ, respectively. An alternative procedure, involving cold soaking, which avoids loss by oxidation and isomerisation but does not result in complete extraction of the pigments, is a modification of the A.O.A.C. method (Thompson and Bickoff, *Brit. Abstr. C*, 1951, 269). In replicate analysis, standard deviations for carotene were $\pm$ 2·97 and $\pm$ 2·65 mg per lb, and for xanthophyll $\pm$ 10·14 and $\pm$ 1·90 mg per lb for the first and second procedures, respectively.

A. A. ELDRIDGE

215. **Determination of free gossypol in mixed feeds.** R. W. Storherr and K. T. Holley (*J. Agric. Food Chem.*, 1954, **2** [14], 745–747).—Butan-g-one - water azeotrope containing aniline extracts a soluble form of gossypol from some meals and feeds in $\simeq$ 10 min., but a further amount of more firmly bound gossypol is extracted for up to 380 hr. In gossypol analyses, a 1-hr. extraction is used for meals and a 2-hr. one for feeds, and analyses are carried out on the filtered solution within 1 hr., as free gossypol is liberated from soluble combined forms. To the azeotrope - aniline solution (2 ml) are added 0·4 per cent. ethanolic phloroglucinol (1 ml) and conc. HCl (2 ml). After 20 min. the mixture is diluted to 25 ml with ethanol or *iso*propanol, and the optical density is measured at 550 mμ. Results obtained with meals and feeds are compared with those obtained by the *p*-anisidine method.

C. E. SEARLE

216. **Electrophoresis of some neutral polysaccharides.** D. H. Northcote (*Biochem. J.*, 1954, **58** [3], 353–358).—An electrophoretic method is described for analysis of neutral polysaccharides. It is of general application; electrophoresis of a 1 per cent. solution of the soluble polysaccharide in 0·05 *M* borate buffer is carried out at 0° C. In this buffer (pH 9·2), the polysaccharides migrate towards the anode. In certain instances a 1 per cent. solution in glycine buffer (pH 9·2) or in barbitone buffer (pH 9·2) is used. The boundaries are more stable in glycine buffer, but the mobilities are much lower than in borate buffer. Borate buffer is essential for satisfactory electrophoretic analysis even with polysaccharides that contain only trans vicinal hydroxyl groups; this indicates a combination of borate with this type of polysaccharide. Polysaccharides different in size, shape and structure, but composed of the same sugar units, can be differentiated electrophoretically. Ivory-nut mannan and rabbit-liver glycogen appear to be homogeneous electrophoretically; the two main sol. polysaccharides obtained from yeast are glycogen and mannan, and these are clearly separated by electrophoretic analysis, but the yeast glycogen (and starches) show presence of at least two components. The method should be useful in a preliminary study of a mixture of polysaccharide material such as that obtained from many plant products and from cell structures, such as cell walls.

J. N. ASHLEY

217. **The analysis of flavones. V. The characterisation of the position of substitution on the phenyl side chain by some reactions of the reduction products.** L. Hörhammer and K. H. Müller (*Arch. Pharm., Berlin*, 1954, **287** [7], 376–380).—The red colour produced by the reduction of polyoxyflavones with Mg in HCl changes to red-violet or blue on the addition of Na acetate or alkalis only if there is a free hydroxyl group on the C_4 carbon atom. A blue colour with a drop of $FeCl_3$ solution is given by reduced flavones having two ortho hydroxyl groups. The flavone is reduced as above and extracted from excess of Mg and HCl with pentanol before the $FeCl_3$ solution is added. Only a few hundred μg are required for these reactions.

P. S. STROSS

218. **Spectrographic analysis of exchangeable cations in soils.** F. Burriel-Martí, S. Jiménez Gómez and C. Alvarez (*An. Soc. Esp. Fis. Quím., B*, 1954, **50** [7–8], 663–672).—In the spectrographic determination of Na, K, Mg, Ca, Pb and Mn in the concentrations found in Spanish soils, when Ca is not to be determined, 270 to 430 mμ is the best region. Calciferous soils are best determined in the region 300 to 625 mμ, but Mg is not determinable in this range.

T. R. MANLEY

219. **Rapid method for determination of actual humifying efficiency of organic soil improvers.** J. Pomot and P. Lecat (*Compt. Rend. Acad. Agric., France*, 1954, **40** [13], 494–499).—The test depends on the measurement by the ^{32}P technique of the effect of organic matter in a non-nutrient "soil" on ^{32}P-uptake by rye seedlings. Rye is sown in 750-ml pots (40 seeds per pot, in two concentric circles round a central tube), each containing a mixture of sand (250 g) and granulated silica (100 g, 3 to 4 mm in diameter), with a drainage layer of silica granules (50 g). The mixture in each of the test-pots is incorporated with 350 mg of the organic matter under test, and "blank" pots are set up without organic matter. After addition to each pot, through the central tube, of 60 ml of aq. 0·1 per cent. w/v $NH_4H_2{}^{32}PO_4$, the pots are incubated at 20° C ($\pm$ 1° C) under constant illumination during 8 days; the average radioactivities of the part of the seedlings (divested of the coleoptile) exposed to the air, are then determined by means of a Geiger counter. Determinations on sets of 6 seedlings (equally divided between the outer and inner circles) from 2 or 3 pots (blanks as well as tests) are necessary and sufficient. The humification factor for the organic matter is calculated from the formula: $1000(H-T)/T$, where H and T are the average counts per min. for the seedlings from the tests and blanks, respectively.

P. S. ARUP

220. **The determination of parathion.** S. Schomberg (*Chim. et Ind.*, 1954, **72** [3], 444–446).—Current methods for determination of parathion are critically discussed, and a proposed method is based on the determination of organic phosphorus. The sample is extracted with light petroleum for 3 or 4 hr., and the extract is washed 3 times with 10-ml portions of water. Alcoholic KOH (20 ml) is added and after 15 min. the mixture is refluxed for 15 min. on a water-bath, made up to volume and inorganic P_2O_5 is determined in an aliquot. The remainder of the solution is evaporated to dryness and after cooling oxidised with 10 ml of a (7 + 5) mixture by vol. of $HClO_4$ (sp. gr. 1·54) and HNO_3 (sp. gr. 1·40). The oxidation is repeated twice. After cooling, 10 ml of water and 15 ml of H_2SO_4 - HNO_3 (Lorentz) are added and, after heating, the phosphate is precipitated with nitro-molybdate. The precipitate is analysed by dissolving it in standard KOH or by the micro Kjeldahl procedure. To determine if other organic phosphates are present, investigation of the P to S ratio in an aliquot is suggested.

E. J. H. BIRCH

221. **Biological screening test for chlorinated insecticides.** B. Davidow and F. J. Sabatino (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 902–905).—

The sensitivity of goldfish to DDT, Lindane, Heptachlor, Toxaphene, Dieldrin, Aldrin, Chlordane, Methoxychlor and Dilan has been determined approximately by observation of the onset of convulsions and loss of equilibrium. *Daphnia magna* may also prove to be a suitable test organism. For the examination of foods, a purée is distilled with steam, and goldfish placed in the distillate are observed for 3 hr. A. A. ELDRIDGE

See also Abstract 64.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

222. **Volumetric glassware. Scientific aspects of design and accuracy.** D. S. I. R. (N.P.L. Notes Appl. Sci., No. 6, 21 pp.).—The basic scientific information that must be taken into account in the preparation of standards for volumetric glassware is presented, together with the principal features of standardisation of general applicability. The topics covered include units of vol., standard temp., construction and verification of apparatus, determination of capacity by the gravimetric method, dependence of vol. delivered on delivery procedure, drainage time and delivery of liquids other than water.
J. M. JACOBS

223. **A continuously acting cloud chamber.** H. J. C. Tendeloo and J. W. van Wijk (*Chem. Weekbl.*, 1954, **50** [32], 555–556).—The apparatus described consists essentially of a bell-jar (with tubulure) resting on the loose cover of an insulated tin box and containing, in the upper part, a warm-water tank suspended by means of a brass tube passing through a rubber stopper in the tubulure. Felt attached to the bottom of the tank is soaked with 50 per cent. ethanol, and solid CO_2 is pressed by means of a spring against the lower side of the tin cover. Radioactive material is introduced by means of a suitable attachment on the end of a bakelite rod passing through the brass tube. A potential of 200 V is maintained between the brass tube and the tin box. P. S. ARUP

224. **The automatic counting of red blood cells.** E. H. Cooke-Yarborough and R. E. Whyard (*Brit. J. Appl. Phys.*, 1954, Suppl. No. 3, s147–s156).—The automatic blood-cell counter described and illustrated comprises a microscope on the stage of which a glass haemacytometer (40 by 20 by 0·1 mm) is placed. The haemacytometer contains a dil. suspension of blood in 0·1 per cent. iodine in isotonic KI plus 30 ml per litre of 1 per cent. eosin. The image of the glass floor (on which the blood-cells have settled) is focused on to a specially shaped aperture, which defines the area on the floor at any instant by means of a photomultiplier situated behind the aperture. Linear scanning is effected by a suitable mechanical drive to the stage, and electrical pulses from the photomultiplier are counted by an electronic scaler and a recorder. The errors inherent in the method are discussed, and it is explained how they may be minimised by use of a special electronic circuit (shown), by careful choice of parameters and by adoption of an adjustable single or double aperture. One aperture scans strips of width, say W, whilst the other (of same area) scans pairs of strips each of width $0{\cdot}5\ W$. The results reported show that reproducible values (standard deviation $\pm$ 3 per cent.) can be obtained which are largely independent of variations in light intensity, focus, cell-size, amplifier and photocell sensitivity. About 100 strips per min. can be scanned, successive runs with the two apertures taking $\simeq$ 4 min. Errors are mainly due to statistical uncertainties and to the method of preparation of the specimen. W. J. BAKER

225. **A mechanical wet-sieve testing method.** J. J. Kobliska and H. J. Rodenberger (*Bull., A.S.T.M.*, 1954, No. 200, 46–47).—The standard apparatus and procedure described eliminate most of the causes of variable results for the wet sieving of powders, *e.g.*, presence of water-insoluble pigments, and decreases by about three-quarters the time needed to make the test. The machine consists of a rotating platform on which a sieve is placed. A flattened stream of water, or other liquid, is directed on to the sieve to cover one-half of the diameter of the rotating sieve and at such an angle that the particles pass under the sheet of water at each revolution of the sieve. Rotation of the sieve and flow of water are accurately controlled for a specified time. W. J. BAKER

226. **A photo-electric apparatus for assessing dust samples.** D. G. Beadle (*J. Chem. Metall. Min. Soc. S. Afr.*, 1954, **55** [2], 30–39).—The theory, construction, operation, performance and calibration of a photo-electric apparatus for assessing dust samples are described. The advantages of the apparatus over the usual microscopic method include speed of operation, consistency of measurement, elimination of the human error and measurement of the surface area instead of particle number concentration. It is designed specifically for assessing samples taken with the modified thermal precipitator (Kitto and Beadle, *J. Chem. Metall. Min. Soc. S. Afr.*, 1952, **52**, 284). G. C. JONES

227. **Automatic coulometric titration of acids.** J. J. Lingane (*Anal. Chim. Acta*, 1954, **11** [3], 283–288).—An electrode assembly is described for the titration of acids by the constant-current coulometric technique. The generator electrodes comprise a platinum cathode and a silver - silver bromide anode. The increase in pH resulting from the reduction of hydrogen ions at the cathode is indicated by a glass electrode in conjunction with a Beckman automatic titrator, which automatically interrupts the generating current when the equivalence-point pH is reached. Quantities of HCl $\simeq 1{\cdot}2 \times 10^{-4}$ mole in 50 ml are titrated with a mean error of $-0{\cdot}07$ per cent.; average deviation from the mean is $\pm$ 0·15 per cent. W. C. JOHNSON

228. **The purification and properties of horse-liver esterase.** [**Apparatus and technique.**] J. Burch (*Biochem. J.*, 1954, **58** [3], 415–426).—An apparatus is described for continuous titration of the acid formed during enzymic hydrolysis of esters. It is designed for use with horse-liver esterase; it measures low reaction rates and requires only very small amounts of substrate. The liberated acid is titrated continuously, and glass electrodes are used for pH control. Changes in electrode potential are detected with $\simeq$ 10 times greater sensitivity than is possible with the usual pH meters. The apparatus described detects changes in concn. of acid $< 5 \times 10^{-7}\ M$.
J. N. ASHLEY

229. **Apparatus for continuously measuring a constituent of a fluid.** E. K. von Brand (Brit. Pat. 715,769. Date Appl. 3.11.51).—The fluid under analysis is passed through a continuously-moving filter-tape strip and the impurities deposited on it are estimated by the colour produced after chemical treatment. G. SKIRROW

230. **Apparatus for dispensing measured quantities of liquids.** National Research Development Corporation (Brit. Pat. 712,876. Date Appl. 4.2.52).—A pipette is fastened to the end of an arcuate tube partly filled with liquid, the other end of the tube being open to the atmosphere. Rotation of the arcuate tube, when the pipette is partly immersed in liquid, causes liquid to flow into the pipette.
G. SKIRROW

231. **A simple inexpensive [chromatographic] fraction collector.** V. Claassen and J. van der Sluis (*Chem. Weekbl.*, 1954, **50** [31], 535–536).—A circular collecting-tube stand, the base of which carries a series of downward-pointing pegs, is kept under rotary tension, movement being checked by the perpendicular pressure of one of the pegs against the surface of one or other of two revolving (1 rev. per hr.) discs (1 cm apart) and momentarily permitted by the successive passage of the pegs through suitably spaced radial slots in the discs. The relative positions of the slots in the two discs are set to give a rotation of the stand through $\simeq 30°$, and the collecting intervals can be adjusted by means of a simple mechanism to 15, 30 or 60 min. P. S. ARUP

232. **Gas analysis apparatus.** K. T. Kalle (Brit. Pat. 714,898. Date Appl. 22.9.52).—An apparatus is described for indicating the amount of CO_2 in combustion gases. A fluid jet pump intimately mixes the gas with the absorbing liquid and a centrifugal pump separates the unabsorbed gas from the liquid. G. SKIRROW

233. **Semi-micro Orsat gas analyser.** J. J. McMullen and O. J. Stark (*J. Ass. Off. Agric. Chem.*, 1954, **37** [3], 856–860).—The apparatus described and illustrated is suitable for the determination of O and CO_2 with an accuracy of $\pm$ 0·2 per cent. in 1 to 10 ml of headspace gas above packed milk powder. The gas burette is of the Van Slyke type.
A. A. ELDRIDGE

234. **New anemometer with adjustable range for low rates of flow.** F. Wenger (*Anal. Chim. Acta*, 1954, **11** [3], 229–232).—A flowmeter for gases is described. The pressure drop between the ends of a glass capillary is indicated by a water-filled U-tube manometer. The effective cross-section of the capillary can be varied by adjusting the position of a tapered glass rod that passes through it; in this way the range of the meter can be altered. A linear relationship between rate of flow and pressure difference is obtained for rates of flow up to 6 ml of gas per sec. W. C. JOHNSON

235. **A nitrous fume [nitrogen dioxide] absorptiometer.** D. V. Sinkinson (*Chem. & Ind.*, 1954, [33], 1003–1004).—A simple photo-electric absorptiometer is described for the determination of NO_2; it is based on the light absorption of the NO_2. The limit of detection is 5 μg per litre and the technique is specific for NO_2 in the absence of other coloured gases. R. J. MAGEE

236. **Simple Kjeldahl distillation apparatus.** D. W. Skidmore (*Ind. Chem.*, 1954, **30**, 386).—The apparatus, constructed from Pyrex glass and provided with ground-glass joints, comprises a 1½-litre steam generator with electric heater, a double-walled steam-distillation chamber, a condenser and a receiver. In micro-scale determinations, $\simeq$ 6 ml of 0·2 *M* solution are used. D. R. PECK

237. **Simple ebulliometric method with the aid of foaming substances. I. Water as solvent.** C. N. Polydoropoulos (*Chem. & Ind.*, 1954, [33], 1000–1002).—A description is given of the construction and use of a simple ebulliometric apparatus. The bulb of the Beckmann thermometer used is situated inside a piece of glass tubing. Over this bulb an equilibrium mixture of solution and vapour circulates, helped by the addition of a trace of a foaming substance (Na stearate, fish glue, saponin, Teepol) to the solution. Constant thermometer readings with a precision of $\pm$ 0·001° C are claimed.
R. J. MAGEE

238. **Modified liquid - liquid extractor for routine oil analysis.** C. R. Buerki and K. E. Holt (*J. Amer. Oil Chem. Soc.*, 1954, **31** [8], 335–338).—The continuous liquid - liquid extractor for routine oil analysis described and illustrated has a magnetic stirring motor. Modifications to the standard procedures are suggested for the determination of total fatty acids, oxidised acids and unsaponifiable matter. Accuracy is improved because handling is limited, and no emulsion difficulties are encountered during extraction; the enclosed system minimises health and fire hazards. D. BAILEY

239. **Ultrasonic liquid level indicator systems** R. L. Rod (*Electronics*, 1954, **27**, 156–161).—A transducer driven by a pulsed oscillator (400 kilocycles per sec.) transmits signals upward from the bottom of the tank, and receives echoes from the surface or any interfaces between immiscible liquids. The pulse triggers time-interval measuring circuits and the elapsed time is displayed on decade counters. The accuracy is $\simeq$ 0·01 ft. in 50 ft. Manual calibration methods are described as well as an automatic system having a second transducer, which receives echoes from protuberances at known depths inside a still well. This system also compensates for changes in the velocity of sound by producing an error voltage that changes the frequency of the timing oscillator. S.C.I. ABSTR.

240. **Pressure measurement with the Pirani gauge.** K.-D. Mielenz (*Z. angew., Phys.*, 1954, **6** [3], 101–104).—A standard form and arrangement of gauge is described. The filament consists of 1 metre of 20-μ platinum wire. An expression for the sensitivity is derived and it is shown that by proper choice of arrangement and, particularly the wall and filament temp., max. sensitivity can be obtained. This has been verified experimentally, and a gauge has been constructed for which experimental values are tabulated and shown graphically; pressures down to $1{\cdot}7 \times 10^{-5}$ mm of Hg can be measured.
S.C.I. ABSTR.

241. **Sublimation apparatus for rapid separations.** L. N. Pino and W. S. Zehrung (*J. Chem. Educ.*, 1954, **31** [9], 476).—For the separation of reasonably heat-stable ortho and para isomers, sublimation is more rapid than steam distillation or fractional crystallisation. The apparatus illustrated comprises a sublimation chamber, 52 cm × 2·6 cm in diam., fitted inside a heating tube, 61 cm × 4·2 cm, which is wound with 25 ft. of No. 22 Chromel wire as the heating coil, to which the power input is governed by a variable auto-transformer. One end of the sublimation tube just fits into a condenser tube, 72 cm × 3·2 cm, and at the other end is fitted an inlet tube, 5·8 cm × 2 cm, containing Drierite between two asbestos plugs. An aspirator connected to the exit end of the condenser chamber provides an air current to transport the vapours of the sublimate. After a separation, sublimate and residue can be extracted separately with solvent and recrystallised. Mixtures (20 to 60 g) of 2-amino-3-nitropyridine and 2-amino-5-nitropyridine were

rapidly and almost quantitatively separated at 120° C by sublimation. G. HELMS

Optical

242. **Double-chamber electrode for spectrochemical determination of chlorine and other halogens.** L. H. de Paiva Azevedo, A. W. Specht and R. S. Harner (*Spectrochim. Acta*, 1954, **6** [4], 331–333).—A double-chamber graphite electrode suitable for d.c. arc determination of halogens by means of the alkaline-earth halide bands is described. An upper chamber holds the alkaline-earth compound and the halogen compound is in an interconnected lower chamber. By this arrangement there is an abundance of alkaline earths in the arc by the time the halogen is volatilised from the lower chamber, thus giving max. emission of the alkaline-earth halide bands. A. J. MEE

243. **An improved slit-varying mechanism for monochromators.** Beckman Instruments, Inc. (Brit. Pat. 716,057. Date Appl. 20.2.52).—Specifications are given of a variable-slit mechanism comprising a number of slits each including a movable and a fixed jaw. G. SKIRROW

244. **Mechanism for uniform wavelength scan in spectrometers.** C. A. Parsons & Co. Ltd. (Brit. Pat. 713,288. Date Appl. 18.6.52).—The device described permits concentration on one or two narrow spectral ranges for the isolation of particular absorption bands. G. SKIRROW

245. **Apparatus for microwave spectroscopy.** M. W. P. Strandberg, H. R. Johnson and J. R. Eshbach (*Rev. Sci. Instrum.*, 1954, **25** [8], 776–792).—Design considerations are given for a Video microwave spectrograph with Stark modulation and a crystal or bolometer detector. G. SKIRROW

246. **A high-temperature microwave spectrometer.** M. L. Stitch, A. Honig and C. H. Townes (*Rev. Sci. Instrum.*, 1954, **25** [8], 759–764).—Pure rotational transition lines of KCl, $CaCl_2$ and NaCl and unexplained rotation spectra from samples of titanium and indium chlorides have been observed at temp. of 300° to 775° C in a microwave spectrometer designed for measurement at gas temperatures as high as 1000° C. Microwaves pass through a 5-ft. nickel absorption cell. Absorption lines are modulated by Stark effect to give sensitive detection. G. SKIRROW

247. **A rapid and sensitive recording spectrophotometer for the visible and ultra-violet region. I. Description and performance. II. Electronic circuits.** Chia-Chih Yang and V. Legallais (*Rev. Sci. Instrum.*, 1954, **25** [8], 801–813).—The design, construction and circuit details are given of a double-beam recording spectrophotometer for rapidly obtaining spectra of labile intermediates in biochemical reactions. The ratio of light intensities in two optical paths (both obtained from a single monochromator by a chopping mirror) is measured and converted electronically into units of optical density. A servo correcting system permits linear recording at a max. rate of 6 mμ per sec. An overall accuracy of about 2 per cent. is claimed. G. SKIRROW

Thermal

248. **Ardonox: a new Ardometer [radiation pyrometer].** F. Lieneweg and A. Schaller (*Siemens-Z.*, 1954, **28**, 67–73.)—Unlike the older form, the new instrument works almost entirely within the i.r. range, and has an optical system of the Féry type. The concave mirror has an evaporated aluminium reflecting film with a protecting layer of quartz. Entry of dust and air-currents are prevented by a front window of synthetic film (0·1-mm thick), which is transparent far into the i. r.; radiation loss at room temp. due to the film is only 30 per cent. The radiation-sensitive element is a thermopile of 23 Nichrome - Constantan couples; the output is 1 mV at 100°, 5 mV at 300°, 10 mV at 400° and 30 mV at 600° C. Ranges obtainable are: 500° to 600° C with a directly-connected indicator or (chopper-bar) recorder; 400° C with a d.c. amplifier of the a.c.- modulated type; 400° C with an (electronic) potentiometric recorder; 150° C with a light-beam galvanometer; and 50° C with a photoelectric d.c. amplifier. Typical scale-shapes are given for ranges of 0° to 50°, 20° to 75°, 50° to 300° and 100° to 600° C. Ambient temp. compensation is effected by a temp.-sensitive resistor in parallel with the thermopile, and a series-connected battery-fed resistance bridge with three fixed and one temp.-sensitive arms. Min. radiator source diameter is 9 cm at 10 cm distance, 19 cm at 100 cm, and 52 cm at 400 cm. S.C.I. ABSTR.

Electrical

249. **Experiments with platinised platinum electrode.** T. de Vries (*Chem. Weekbl.*, 1954, **50** [31], 533–535).—In potentiometric titrations of $Fe^{\cdot\cdot}$ with $Ce(SO_4)_2$, in presence of atmospheric oxygen and at concn. of $Fe^{\cdot\cdot} < 0{\cdot}001\ M$, the platinised platinum electrode may function partly as an oxygen electrode. Regeneration may be effected by electrolysis of dil. H_2SO_4 in which the platinised electrode is used as cathode. P. S. ARUP

250. **A photo-electric densitometer for paper-electrophoresis.** N. Cerf (*Chim. Anal.*, 1954, **36** [10], 268–271).—A complete photo-electric apparatus that permits accurate densitometric measurements of coloured bands obtained on filter-paper by electrophoresis or chromatography is described. Bands ($\simeq$ 40 mm) are examined by moving the paper at a rate of 0·7 mm per sec., recordings being made every 3 sec. The resulting density curves are automatically registered. D. R. GLASSON

251. **New high-frequency mass spectrograph.** J. Kramer and J. B. Le Poole (*Ind. Chim. Belge*, 1954, **19** [7], 705–706).—In the time-of-travel mass spectrograph described and illustrated, the ion beam is deflected by a rotating electric field of constant strength, the resultant conical beam being focused on to a second identical deflector by an electrostatic lens. The deflectors comprise 20 specially arranged electrode rods energised by high-frequency generators ($\simeq$ 100 kilocycles) to give a 90° phase-difference; a point ion source is essential. The second electric field is rotated through an angle equiv. to the time of travel of the ions, that is, proportional to $m^{0{\cdot}5}$, where m = ion mass. The final deflection is the sum of two equal rotating vectors at this angle, and the mass spectrum is a system of concentric circles. Since no beam-splitting is required, improved resolution and higher intensity are attained than with the usual instrument. W. J. BAKER

252. **Process for detecting impurities in inert gases.** O. H. Drager (Brit. Pat. 716,015. Date Appl. 17.11.52).—The gases are conducted through an electrolytic rectifier; from the leakage current of the rectifier the extent of impurities is determined. G. SKIRROW

See also Abstract 3.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångstrom unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
critical	crit.
crystalline, crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c.p.s.
decompos -ing, -ition	(decomp.)
density	ρ
density, relative	d or wt. per ml
derivative	deriv.
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
electron-volt	eV
equivalent	equiv.
experiment	expt.
foot, feet	ft.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
horse-power	h.p.
hour	hr.
hydrogen ion concentration	$[H^{\cdot}]$
hydrogen ion exponent	pH
inch	in.
infra-red	i.r.
insoluble	insol.
kilogram	kg
kilovolt	kV
kilowatt	kW
maxim -um, -a	max.
melting-point	m.p.
mitro-curie	μC
microgram	μg
micro-litre	μl
micron	μ
milliampere	mA
milli-curie	mC
milligram	mg
millilitre	ml
millimetre	mm
millimicron	mμ
millivolt	mV
minimum	min.
minute (time)	min.
molar (concentration)	M
molecul -e, -ar	mol.
normal (concentration)	N
number	no.
observed	(obs.)
ounce	oz
part	pt.
patent	pat.
parts per million	p.p.m.
per cent. wt. in wt.	per cent. w/w
per cent. wt. in vol.	per cent. w/v
per cent. vol. in vol.	per cent. v/v
potential difference	p.d.
pound	lb
precipitate	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	$n\lambda^t$
relative humidity	R.H.
revolutions per minute	r.p.m
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]\lambda^t$
square centimetre	sq. cm
standard temperature and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition the following symbols are used—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\backsimeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

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